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ABSTRACTS OF CURRENT PUBLISHED INFORMATION ON NICKEL AND ITS ALLOYS

GENERAL

Standard Hardness Conversion Tables

AMER. SOC. TESTING MATERIALS: 'Standard Hardness Conversion Tables for Metals: Relation between Brinell Hardness, Diamond Pyramid Hardness, Rockwell Hardness, and Rockwell Superficial Hardness.'

A.S.T.M. Designation E 140–58.
A.S.T.M. Standards, 1958, *Pt. II*, pp. 1323–30.

These tables represent a revision and consolidation of the Standard Hardness Conversion Tables for Steel (*E* 48–47), for Nickel and High-Nickel Alloys (*E* 93–53), and for Cartridge Brass (*E* 33–42), which are now discontinued.

The new consolidated Standard covers conversion relationships for the following materials:

(1) *Carbon, Alloy and Tool Steels*, in the as-forged, annealed, normalized, and quenched-and-tempered conditions, provided that they are homogeneous.

(2) *Nickel and High-Nickel Alloys* (nickel over 50 per cent.), relating particularly to the following types of alloy: nickel-aluminium-silicon specimens finished to commercial mill standards for hardness testing, covering the entire range of these alloys from the annealed to the heavily cold-worked or age-hardened conditions, or both, including their intermediate conditions.

(3) *Cartridge Brass*. The specification lays down the standard methods by which the hardness readings shall be obtained for conversion, and deals with the principles which are to be adopted in conversion, the factors affecting validity of conversion, and the significance of hardness tests.

Diffusion in the Solid State

C. E. BIRCHENALL: 'The Mechanism of Diffusion in the Solid State.'

Metallurgical Reviews, 1958, vol. 3, No. 11, pp. 235–77.

Some indication of the scope and intention of the review may be obtained from the following excerpt from the author's introduction: 'The great volume of research now being done on solid-state diffusion, resulting in more than one hundred papers per year, gives some idea of the importance which is attached to understanding the mechanisms by which, and the rates at which, atoms move with respect to their neighbours in crystalline substances. In the limited space available for this review, it is impossible to give a complete bibliography, or to discuss all the interesting problems at present under investigation. Fortunately, a bibliography, nearly complete up to the end of 1956, can be quickly compiled from the many recent monographs, symposia and review articles. This account refers only to the outstanding papers bearing on each topic discussed.'

The review, which is supported by a *bibliography* of 192 items, includes sections on:

The Diffusion Coefficient

Diffusion Mechanisms in Metals
(The Kirkendall Effect; temperature dependence of diffusivity; activation energy; frequency factor)

Diffusion Mechanisms in Ionic Crystals
(Cation diffusion in oxides with the NaCl structure; spinel oxides)

Diffusion Mechanisms in Covalent Crystals

Boundary and Surface Diffusion

Reference is included to nickel-containing alloys which illustrate some aspects of diffusion, and an appendix relating to self-diffusion in 23 solid elements gives data on nickel.

Terms Used in Powder Metallurgy

'Standard Definitions of Terms Used in Powder Metallurgy.'

A.S.T.M. Designation 243–58.
A.S.T.M. Standards, 1958, *Part II*, pp. 1025–9.

Contains definitions of 115 terms.

See also

'International Powder Metallurgy Glossary.'
Metal Powder Report, 1958, vol. 13, Oct., insert.

For the purpose of assisting the development of powder metallurgy in various countries of the world, it is proposed to publish, in future issues of *Metal Powder Report*, a glossary or set of definitions (in a variety of languages) of all important terms commonly used in powder metallurgy.

Versions in the respective languages (including Afrikaans, Arabic, Chinese, Hebrew, Hungarian, Japanese, Turkish, Russian, etc.) are being prepared by scientists and technologists in the various countries.

This issue of *Metal Powder Report* contains the English version of the glossary, upon which all others are being based. The definitions are founded on those drawn up by the American Society for Testing Materials (*see* preceding abstract) and incorporates also modifications suggested by other authorities.

Bonding of Cermets to Metals

G. R. VAN HOUTEN: 'A Survey of the Bonding of Cermets to Metals.'

Welding Jnl., 1958, vol. 37, Dec., pp. 558s–69s.

The brazing of cermets to metals is complicated by the large differences in thermal coefficients of expansion of the two types of material and by the difficulty in wetting of cermet surfaces by the brazing metals. The need for brazes which are strong at high temperatures (and therefore melt at even higher temperatures) further increases the problem of differential expansion. This review, although originating in a specific problem (joining of a large nickel-bonded titanium-carbide cermet to a main metallic structure) covers all aspects of ceramic/metal bonding.

The correlated information presented is based on 168 *selected items of technical and patent literature*. It is arranged under the following classifications:

Introductory Section

The Nature of the Problem involved
The History of Ceramic/Metal Seals

Literature Survey

Cermets
Definition, evolution, properties (general; wettability)

Filler Materials for Cermet-to-Metal Brazing
Conventional materials; zinc-rich and copper-rich braze materials; miscellaneous braze materials

General Brazing Procedures
The problem of wettability; joint design; joint strength; joint soundness; general considerations

Procedures for Brazing Cemented Carbides
Problems peculiar to carbide brazing; solution of carbide-brazing problems (by elimination of brazed joints, by use of thick brazed joints, sandwich braze, electrodeposits; use of materials with matching coefficients of expansion; laminated cermets, modification of joint design; refinement of conventional techniques)

Special Brazing Procedures
Brazing of porous compacts; use of brazing pellets; use of active-metal brazes.
Use of molybdenum-manganese, active-metals, and active-metal-hydrides processes in metal-to-cermet bonding.

Physical Testing of Brazes

Consideration of the wide range of literature and experience on which the survey is based leads the author to the conclusion that the use of hydride, active-metals and molybdenum-manganese surface-preparation techniques, together with high-temperature braze metals and base metals with matching thermal coefficients of expansion, appears promising.

NICKEL

High-Purity Nickel

The following statement was published in *Chemical Week*, Oct. 11, 1958, p. 103.

'Hyperpure Nickel to be Available in Pilot-Lot Quantities.

'Hyperpure nickel will soon be available in pilot-lot quantities from U.S. Bureau of Mines (Salt Lake City). Improved equipment and extraction techniques have yielded laboratory quantities of nickel 'containing less than a few parts per million of cobalt'. The scaled-up pilot facility will have a capacity exceeding 1 ton/day of feed, equivalent to about 20 lb. of hyperpure nickel. Key to the high purity is a solvent extraction process based on amine extraction of hydrochloric-acid solutions of nickel and cobalt. Tri-iso-octylamine has been found particularly effective. Cobalt recovered as a co-product of the hyperpure nickel is also extremely pure, contains no chemically detectable nickel. The new pilot plant will turn out about 1 lb./day of cobalt.'

Forms of Nickel available as Alloying Additions for Steels

See abstract on p. 48.

Specific Heat of Iron and Nickel

S. VALENTINER: 'The Specific Heat of Iron and Nickel.'

Archiv f.d. Eisenhüttenwesen, 1958, vol. 29, Nov., pp. 685–8.

In recent years a great deal of work has been carried out on the experimental determination of the true specific heats of nickel and iron, as a function of temperature. The values obtained sometimes conflict, and, where this is so, the author advises recourse to data derived theoretically from the results of experiments in various other fields. Extrapolation of such data, to cover a wide range of temperature, provides a means of comparison which will serve as a guide to the likely accuracy of the experimental values.

In the present paper this technique is applied to selection, from the available data, of the true specific heats of iron and nickel over the temperature range 0°C. to the Curie point. The experimental results and theoretical data which form the basis of comparison are presented in tabular and graphical form. The values obtained are used to calculate the spontaneous magnetization of the two metals at high temperatures.

The true specific heats of nickel at various temperatures are given on page 39.

Temperature °C.	Specific Heat Calories/g./°C.
20	0·1060
100	0·1113
160	0·1177
220	0·1251
300	0·1358

Influence of Nickel on the Solubility of Nitrogen in Liquid Iron

H. SCHENCK, M. G. FROHBERG and H. GRAF: 'Investigation of the Effect of other Elements on the Equilibrium Solubility of Nitrogen in Iron (I).'

Archiv f.d. Eisenhüttenwesen, 1958, vol. 29, Nov., pp. 673-6.

In view of the deleterious effects which nitrogen has on the properties of iron and its alloys, numerous experiments (to which reference is made) have been conducted to determine the susceptibility of molten iron to nitrogen pick-up. The investigation described here extends these experiments to determination of the influence of a third element, on the degree to which nitrogen is soluble in molten iron.

Melts containing various amounts of antimony, arsenic, cobalt, copper, molybdenum, nickel, oxygen, sulphur and tin were exposed, at 1600°C. and for a period long enough to ensure equilibrium conditions, to a jet of nitrogen under a pressure of 1 atm. In the case of the cobalt and molybdenum additions the nitrogen was also introduced at 1550° and 1650°C. Specimens were then quenched and their nitrogen content was determined.

The results showed that, with the exception of molybdenum, all the addition elements decreased the solubility of nitrogen in liquid iron. Molybdenum alone had the reverse effect. Data derived from the literature and the results of the present experiments are tabulated, comparing the effect, on nitrogen solubility, of various elements. In addition to those studied by the author, values are given for carbon, phosphorus, manganese, silicon, chromium and vanadium.

Diffusion in the Solid State

See abstract on p. 37.

Corrosion of Nickel in Sulphuric Acid

C. H. PITT and M. E. WADSWORTH: 'Kinetic Study of the Corrosion of Nickel in Sulphuric Acid.'

U.S. Atomic Energy Commission, Publn. NP-6208, 1957; 24 pp. *P.B.* 132477.

Corrosion of a metal in an aqueous solution may occur galvanically, or directly by electron transfer (oxidation-reduction) at a single site on the surface. The aim of the work described was to investigate the latter process in relation to corrosion of nickel in sulphuric-acid solutions, in the temperature range 75°–125°C. and under an oxygen atmosphere at a partial pressure ranging from 0 to 600 p.s.i. (0–0·26 t.s.i.; 0–0·4 kg./mm.²).

The influence, on the corrosion rate, of the following factors was determined by a series of experiments (run for periods between 30 and 70 minutes), in which each variable was changed in turn: agitation of the solution, time, temperature, oxygen concentration, concentration of species furnished by sulphuric acid, surface area of the sample, concentration of products. The kinetics of the corrosion process were studied by determination of the amount of nickel ion in solution at specific intervals of time.

The corrosion rate was found to vary linearly with time and to increase directly with increase in oxygen partial pressure. No appreciable corrosion was observed in the absence of oxygen, and the corrosion rate was not affected by agitation above a certain speed of stirring.

These results, and data obtained from varying the concentrations of acid, are considered in relation to the corrosion mechanism involved. It is suggested that corrosion progresses by single-site adsorption of the un-dissociated acid molecule, which in turn reacts with oxygen. Calculation of the number of possible sites indicated that at a given instant a very small percentage of the nickel surface is reactive and that corrosion may nucleate at dislocations and travel concentrically along grain surfaces. The overall reaction is given as

$$2Ni + H_2SO_4 + O_2 + 2H^+ = 2Ni^{++} + SO_4^{=} + 2H_2O.$$

Standard Hardness Conversion Tables

See abstract on p. 37.

Determination of Nickel in Sodium

T. M. FLORENCE: 'Estimation of Traces of Nickel in Sodium Metal.'

Analytica Chimica Acta, 1958, vol. 19, Dec., pp. 548-51.

BRUMMET and HOLLWEG, in 1956, described procedure by which 0·5 per cent. potassium-cyanide solution was used for selective dissolution of nickel from oxidized films on nickel metal and the complex was then analysed spectrophotometrically at 268 mμ. (*Anal. Chem.*, 1956, vol. 28, p. 887; *Nickel Bulletin*, 1956, vol. 29, No. 7, p. 125). The present author describes a study of this method with a view to its potential usefulness for the estimation of traces of nickel in metallic sodium. Interference of iron and chromium is eliminated by precipitation of the hydroxides from homogeneous solution. The method, which is simple and sensitive, is believed to be applicable to a wide range of materials.

Photochemical Properties of Nickel Compounds

K. VEERIAH: 'Photochemical Properties of Nickel Compounds.'

Current Science, 1958, vol. 27, Aug., pp. 298-9.

While nickel compounds are not known to exhibit

pronounced photochemical properties, comparable, for example, to those of compounds of iron, cases of such activity are to be found in the literature. PLOTNIKOV has, for example, reported (*Allgemeine Photochemie*, 2*nd Edn.*, 1936, p. 427) that the green surface of nickel hydroxide darkened upon exposure to light, and BALY (*Photosynthesis*, 1940, p. 96) noted a similar effect with respect to nickel carbonate exposed to sunlight. No attempt has, however, been made to investigate the nature of the chemical change involved in darkening.

In the course of a systematic study of the effect, on solutions of nickel nitrate, of sunlight and light from mercury arc, the present author found that the compound decomposes to yield nickel nitrite, oxygen, and a black solid corresponding to Ni_2O_3. (The course of the reaction was followed by estimation of the nitrite content of the exposed solutions.) The photolysis of nickel nitrate is similar, therefore, to that reported for alkali nitrates, chlorates or perchlorates, which form lower compounds and evolve oxygen. Further investigation showed that addition of an acid, such as acetic acid, retarded photolysis, but that addition of sodium hydroxide had the reverse effect. Green nickelous oxide formed by the addition of sodium hydroxide to nickel nitrate rapidly turned black on exposure to sunlight or ultra-violet light.

Exposure to sunlight, of nickel hydroxide and nickel carbonate precipitated from solutions other than nitrate, chlorate or perchlorate, e.g., from sulphate or chloride solutions, did not result in darkening, but on being mixed with small quantities of nitrate, chlorate or perchlorate, such precipitates did turn dark by photoaction. It is considered significant that Baly's experiments were conducted with nickel carbonate precipitated from nickel nitrate, and it would appear that the darkening of the solids observed by previous investigators was due to the presence, on the particles, of traces of nitrate. Photolysis of nitrate ion, to yield nitrite ion and atomic oxygen, and the combination of atomic oxygen and nickelous oxide, to yield nickelic oxide, is suggested in explanation of the phenomena observed in the cases reported.

Fuller details of the author's work are to be published later.

Nickel-plated Nickel Structures for Resistance to Thermodynamic Heating

See abstract on p. 54

ELECTRODEPOSITION AND OTHER COATING METHODS

A.S.T.M. Specifications for Electrodeposited Coatings

Revisions have recently been introduced into certain A.S.T.M. Standard Specifications: for details see right-hand column.

A.S.T.M. Standards, 1958, *Part II, Non-Ferrous Metals.*

'Electrodeposited Coatings of Nickel and Chromium on Steel.'
*A.S.T.M. Designation A*166–58*T* pp. 933–6.

'Electrodeposited Coatings of Nickel and Chromium on Copper and Copper-base Alloys.'
*A.S.T.M. Designation B*141–58 pp. 937–40.

'Electrodeposited Coatings of Nickel and Chromium on Zinc and Zinc-base Alloys.'
*A.S.T.M. Designation B*142–58 pp. 941–4.

All three specifications are published under the joint aegis of *The American Society for Testing Materials* and *The American Electroplaters' Society*. Requirements are laid down within each specification for various grades of coating to cover thicknesses of plating necessary to provide protection in service conditions of varying severity.

Anodic Behaviour of Nickel in Electroplating Solutions

N. F. MURPHY and B. C. OZA: 'Anodic Behaviour of Nickel: I. Effect of Components of Electrolyte.'
Bull. Virginia Polytechnic Inst., 1958, vol. 51, May; *Engineering Experiment Station Ser.* 125; 18 pp.

Anodic behaviour of metals has recently been the subject of considerable investigation, including studies undertaken for the purpose of (*a*) elucidating the nature and theory of passivity, (*b*) determining the behaviour of electrolytic anodes, (*c*) analysing the mechanism of corrosion of certain alloys, and (*d*) expanding the theoretical laws of electrochemistry.

Several characteristics of nickel have been classified under the term 'passivity'. First, there is the true electrochemical passivity, in which the whole exposed surface of the metal acts as a noble metal. No measurable amount of metal passes into solution when it is made an anode under conditions which stabilize this type of passivity. Second, nickel anodes appear to possess the ability to develop localized passivity, whilst another adjacent portion remains active. This type of behaviour results in high local current density, formation of anode sludges and slimes, and considerable difficulty in obtaining satisfactory cathode deposits or making electrochemical measurements.

Considerable research has been carried out to determine the possibility of preventing formation of anode sludges and slimes by control of the composition of the anode and its metallographic structure. This aspect is not, however, discussed in the present paper, which deals mainly with the influence of composition of solution on overall passivity of nickel anodes, in which condition nickel does not go into solution according to Faraday's law, but, instead, oxygen is evolved. The effect during electroplating is depletion of the solution in nickel ions and changes in pH and composition of the electrolyte.

Following introductory survey of relevant literature,

the authors describe experiments in which the behaviour of nickel anodes was compared with that of platinum (always passive under the conditions used), copper (which can be made passive only under extreme conditions), and iron (selected because its passivity characteristics are already well known). In view of the possibility that the composition of the nickel may influence anodic behaviour, three types of material were included in the tests: (1) 'carbon-free' (C<0·02%) sheet nickel (0·025 in.; 0·625 mm.) thick; (2) commercial-grade nickel containing about 0·5% cobalt; (3) rolled depolarized anodes (99+%) obtained from a commercial supply source. The experimental conditions are fully described and diagrammatically illustrated.

The limits of the activity of the respective anodes were obtained from plots of current density *vs.* anode potential, considered in relation to (1) variations in concentration of the various ions (chloride, sulphate, borate, metal) present in the solutions, and (2) temperature.

The information obtained from these experiments indicates that passivity is a combination of potential and kinetic effects. In electrochemical passivity the condition appears to occur at a particular anode potential, which potential is a resultant of the anode current density. It is considered that, in all probability, in a specific solution a stable passive compound is formed on the surface of the anode under the influence of a certain minimum potential, and as long as the metal-solution potential is exceeded the compound can exist and the anode remains passive.

The composition of the solution environment round the anode is, on the other hand, a function of the velocity of diffusion and velocity of the ions under a potential gradient, at the temperature and concentrations existing. These depend also on the potential (and secondarily on the current density) as well as on concentration, species of ion and composition of the electrolyte.

The results reported indicate that considerable significance should be attached to the effects of alloying elements in nickel anodes, and that further research on this aspect should be carried out.

The passivity behaviour of nickel appears to be closely similar to that characteristic of iron, but the specific conditions which cause it are not necessarily identical with those in which iron becomes passive. Increase in concentration of nickel ions in solution increases the tendency of nickel anodes to become passive: increase in concentration of chloride and sulphate ions has the reverse effect. Passivity limits are widened by raising the temperature. Presence of boric acid in the electrolyte has little effect on the tendency of the anodes to become passive in 2·0*N* nickel solution, but tends to eliminate passivity in a 0·5*N* solution.

Nickel-Plating Solutions and their Applications

H. C. CASTELL: 'Nickel Plating and its Industrial Uses.' *Métaux*, 1958, vol. 33, Sept., pp. 366–76.

The paper constitutes a review of the nickel-plating solutions at present in commercial use. Compositions and operating conditions are summarized, the properties characteristic of coatings deposited from the respective solutions are outlined, and industrial applications for which they have thereby proved suitable are indicated.

The survey contains sections on the following nickel-plating solutions: Watts, sulphate, all-chloride, sulphamate, and bright (nickel-cobalt and organic) types. Processes for electrodeposition of tin-nickel and for electroless deposition of nickel-phosphorus coatings are also discussed.

Production of Patterned Nickel Coatings: The 'Kraq-o-lite' Process

S. S. FREY: 'Pattern Plating with Electrodeposits.' *45th Ann. Tech. Proc., Amer. Electroplaters' Soc.*, 1958, pp. 150–6; disc., p. 171.

In the course of work carried out by the author on compositions for electrocleaning solutions, it was discovered that silicated cleaners having a pH of 12 or lower would, under certain operating conditions, form invisible adherent films and that these films, if not completely removed before bright-nickel plating, could result in dull, etched, or crack-like regions in the deposits. Although means were subsequently found by which the films could be removed or prevented from forming, further investigations were made of the crack-like structure. These studies led to the discovery that the size and number of the cracks were controllable, that the cracks were of laminated form and did not expose the basis metal, and that the 'cracked' deposits exhibited excellent adhesion. Further work was therefore directed to the development of a process which would utilize such a structure as a basis for production of uniformly patterned nickel coatings. The procedure finally evolved, designated the 'Kraq-o-lite' process and covered by the author in *U.S. Patent* 2,780,591, is the subject of the present paper.

'Kraq-o-lite' finishes may be obtained in a variety of colours and pattern effects: copper, gold, or chromium can, for example, be plated over an undercoating of patterned nickel. The basis metal, preferably brass, is first buffed, de-greased, subjected to cathodic treatment in an inhibited cleaner, acid-dipped, and finally made the anode in a specially prepared siliceous solution. Immediately after electrolytic conditioning in this solution the part is subjected to the usual rinse and acid dip, and is then bright-nickel plated to a thickness of 0·1–0·3 mil, which is sufficient to develop the pattern. Further surface coatings, if any, should be deposited from bright solutions, and, if subject to atmospheric tarnishing, should be lacquered prior to use.

The author gives a detailed description of operational procedures. Conditions optimum for the development of the anodic silicated film are outlined, and other sections are concerned with discussion of the influence, on the anodic film, of the silicon-dioxide content of the conditioning solution and of

time and temperature of treatment. The formation and nature of the anodic film are considered, and data are given on nickel-plating solutions which have been shown to be suitable for use as pattern developers.

Influence of the Basis Metal on Stress in Electrodeposited Coatings

J. B. KUSHNER: 'The Rôle of the Basis Metal in the Production of Stressed Electrodeposits.'

45*th Ann. Tech. Proc., Amer. Electroplaters' Soc.*, 1958, pp. 28–32; disc., pp. 158–9.

The author's previous investigations of the causes of residual stress in electrodeposited nickel were described in *Metal Finishing*, 1958, vol. 56, April, May, June and July issues. In that study, which, like the one summarized in the present paper, was based on experimental work supplemented by data available in the literature, an attempt was made to define the relationship between residual stress and such factors as the thickness of the deposit, the cathode film, the temperature and current density used, addition agents, and the basis metal. (The scope of the investigation and the salient findings were referred to in *Nickel Bulletin*, 1958, vol. 31, No. 9–10, pp. 260–1.)

The rôle of the basis metal was studied by determination of the influence of surface cleanliness, surface finish, grain size and the nature of the substrate metal. A theory was advanced which explained the occurrence of residual stress in terms of interference by water, at discontinuities in the surface of the basis metal. The water molecules comprising the entourage of the hydrated or complex metal cation may not be completely removed during deposition, and partial conversion to a hydrated metal oxide will cause a volume change, enhanced by diffusion, out of the depositing metal, of any remaining water molecules. In the case of nickel and the other transition metals the change will be one of contraction, giving rise to tensile stress. According to this theory, the larger the grain size, the fewer will be the dicontinuities present, and the smaller the residual stress. During the initial stages of deposition the surface of the basis metal appears to exert a strong influence on the factors contributing to the formation of stress in the deposit. The stress does not, however, decrease linearly with thickness of the deposit, but falls sharply at a certain distance from the interface, a phenomenon which cannot be attributed to the influence of grain size only. Causes of this and other features of residual stress are discussed in the present paper, in relation to the influence of the basis metal.

The high values of residual stress exhibited by deposits up to about 0·0001 in. (0·0025 mm.) thick is interpreted in terms of differences between the lattice constants of the basis and of the deposited metals: the effects are exemplified by reference to data on nickel deposited on copper and *vice versa*. Difference in lattice constants does not, however, influence the deposit at greater thicknesses, and has little effect on the initial crystal structure of the deposited metal. The factor of importance in this connexion is the size of the grains in the basis metal, which, due to their disorganized atomic structure and higher energy, act as nucleation sites for the deposited coating. The evidence in favour of this theory is reviewed. The influence of grain size on the relationship between deposit thickness and maximum stress is outlined, and a method is described whereby the postulations advanced may be quantitatively verified. The results of preliminary experiments employing this method are given.

Influence of Surface Finish on the Corrosion-Resistance of Nickel Coatings

M. H. JONES and J. ZAJDOWSKI: 'The Influence of the Physical Metallurgy and Mechanical Processing of the Basis Metal on Electroplating.
A.E.S. Research Project No. 14, Part V: Effect of Polishing on the Durability of Nickel-Plated Mild Steel.'

45*th Ann. Tech. Proc., Amer. Electroplaters' Soc.*, 1958, pp. 45–52; disc., pp. 160–1.

Although it has been shown that the surface contour of the basis metal affects the porosity of thin nickel deposits, findings of various investigators with respect to the influence of the same factor on the corrosion-resistance of coatings of commercial thickness have proved conflicting. The discrepancy is thought to result mainly from use, by the various investigators, of steel of different qualities and types as the basis metal: the present authors have found that the quality of the basis metal and its inclusion content significantly influence the degree of corrosion-resistance conferred by the nickel coating (see 44*th Ann. Tech. Proc., Amer. Electroplaters' Soc.*, 1957, p. 53). In the paper now published details are given of research in which statistics were employed 'as a tool in carefully controlled and designed experiments to throw additional light on the problem'.

An aluminium-killed steel of deep-drawing quality was selected as the basis metal, and nine different methods of polishing (representative of commercial and laboratory techniques) were used to evaluate the effects, on corrosion-resistance, of surface finish: commercial dry-belt and greased-belt methods (using alumina abrasives and producing, in each case, fine and coarse finishes); laboratory-type metallographic emery paper (fine and medium), commercial paper (coarse) and diamond abrasives (fine and coarse). The steel was also plated in the as-rolled condition. Full details are given of the finishing techniques employed, the chemical preparation of the surface prior to plating, and the plating procedure. The experiments were designed throughout on a statistical basis. Arrangements for sampling, etc., were such as to minimize errors arising from inhomogeneity of the basis metal and variations in polishing, plating and corrosion testing, and the specimens were uniformly coated, from a Watts solution, with nickel deposits 0·0005 or

0·001 in. (0·0125 or 0·025 mm.) thick. The protection conferred by each coating was assessed by means of the acetic-acid/salt-spray and 'Corrodkote' tests. The corrosion data obtained were subjected to a statistical analysis of variance.

Such assessment of the results showed that the various nickel-plated specimens exhibited significant differences in corrosion-resistance and that these variations correlated with differences in treatment of the basis metal prior to plating. The degree to which the influence of surface treatment was detectable varied, however, with the type of corrosion test and the stage to which corrosion had progressed. Although the acetic-acid/salt-spray test gave useful data, permitting some broad differentiation between the various treatments, the 'Corrodkote' test proved the more discriminating.

With the 0·001-in. (0·025-mm.) deposits closer control could be exerted over the plating and test variables, and the influence of surface finish was more pronounced than in the thinner coatings, particularly as evaluated by the 'Corrodkote' test. The pattern of the results obtained was similar to that for the acetic-acid/salt-spray test, but differences resulting from variation in surface finish of the base metal were discernible in greater detail for different finishes.

The following conclusions are drawn with reference to the influence of the various treatments studied:

(1) For an equivalent surface roughness, commercial greased-belt polishing gives better results than dry-belt polishing.

(2) For both types of commercial polishing, fine finishes (10 μin.) are more beneficial than coarse finishes (40 μin.).

(3) Laboratory-type finishes result in deposits of greater corrosion-resistance than commercial surfaces of similar roughness.

(4) The type of abrasive paper used in laboratory polishing can affect corrosion-resistance.

Effect of Shot-Peening prior to Chromium Plating, on Fatigue Strength of Chromium-Nickel-Molybdenum Steel

See abstract on p. 48.

Thickness of Electrodeposited Nickel Coatings

INTERNATIONAL NICKEL CO., INC.: 'Quality Plating with Thicker Nickel Coatings.'

Issued 1958; 8 pp.

It is pointed out that, over the long period since nickel plating was first introduced as a finish, many facts have been established with regard to variables affecting the quality of the plating and its behaviour in service. One of the factors which have been studied is the influence of thickness of nickel used as final surface coating or as underlay to chromium. Tests under practical, as well as laboratory, conditions all combine to confirm that thicker coatings consistently perform better than thinner ones, and it is with that aspect that the publication is concerned.

In addition to the positive direct evidence which has been obtained in this connexion, indirect proof has been provided by the deterioration in quality which has been observed in coatings produced during the period when the tendency has been to economize in nickel, due to limited supplies. Now that the supply is ample to provide for large-scale use of plating of adequate thickness, platers are reminded of the benefits accruing from compliance with specifications which have been drawn up to cover high-grade nickel coatings.

This recommendation is supported by documented charts embodying data which confirm the value of coatings of adequate thickness, the exact requirements for which are regulated according to the service conditions for which the plated material is to be used. The charts show:

Thickness Requirements of current A.S.T.M./A.E.S. Specifications for Decorative Electrodeposited Coatings.

Effect of Thickness on Protective Value of Buffed Dull Nickel Coatings on Steel.

Effect of Thickness of Nickel (and/or Nickel+Copper) on Protective Value of Decorative (Copper)/Nickel/Chromium Plating under various conditions of atmospheric exposure.

The information given is supported by a *bibliography* of technical papers concerned with the effect of thickness of coating on the durability of decorative plated coatings.

Stripping Electrodeposited Coatings

J. B. MOHLER: 'Stripping Metallic Coatings.'

Metal Finishing, 1958, vol. 56, Dec., pp. 60–3.

The article opens with brief notes on precautions which must be observed in stripping electrodeposited coatings from basis metals and with comment on some stripping methods which are widely applicable. Its main interest consists, however, in 'a listing of recommended methods taken from authoritative sources.'

Details are given of solutions and procedure for stripping (from ferrous and/or non-ferrous basis materials) the following types of metallic coating: brass, cadmium, copper, chromium, gold, lead, nickel, platinum, rhodium, silver, tin and tin alloys, zinc. Recommendations are included also for stripping of anodized, black-oxide and phosphatized coatings.

Nickel-plated Nickel and Copper Structures for Resistance to Thermodynamic Heating

See abstract on p. 54.

NON-FERROUS ALLOYS

Specifications for Nickel-containing Materials

Further to a summary of A.S.T.M. Specifications relating to nickel and nickel alloys appearing in *Nickel Bulletin*, 1959, vol. 32, No. 1, p. 3, attention

is directed below to other schedules dealing with nickel-containing materials which are included in *A.S.T.M. Standards, 1958, Part II, Non-Ferrous Metals.*

A. New Specifications (not previously published in the Standards).

'Nickel-Molybdenum Alloy Plate and Sheet.'
A.S.T.M. Designation B333-58T pp. 573-80.

Covering plate and sheet, in specified conditions, in materials within the following percentage limits of composition: chromium 1·00 max., iron 4·00–6·00, carbon 0·05 max., silicon 1·00 max., cobalt 2·50 max., manganese 1·00 max., vanadium 0·20–0·40, molybdenum 26·00–30·00, phosphorus 0·025 max., sulphur 0·030 max., nickel remainder.

'Nickel-Molybdenum-Chromium Alloy Plate and Sheet.'
A.S.T.M. Designation B334-58T pp. 581-8.

Covering plate and sheet (in the hot-rolled, annealed and de-scaled condition) made in material of the following percentage composition: chromium 14·50–16·50, tungsten 3·00–4·50, iron 4·00–7·00, carbon 0·08 max., silicon 1·00 max., cobalt 2·50 max., manganese 1·00 max., vanadium 0·35 max., molybdenum 15·00–17·00, phosphorus 0·040 max., sulphur 0·030 max., nickel remainder.

'Nickel-Molybdenum Alloy Rod.'
A.S.T.M. Designation B335-58T pp. 620-6.

Covering rod in two conditions: (1) hot-finished, annealed, and de-scaled; (2) hot-finished, annealed and ground or turned. Limiting diameters are laid down. The composition of the material is as specified in *B333-58T*: *see above.*

'Nickel-Molybdenum-Chromium Alloy Rod.'
A.S.T.M. Designation B336-58T pp. 627-33.

Covering rod, in the two conditions specified in *B335-58T* above. Composition called for is as specified in *B334-58T* above.

'Nickel - Molybdenum and Nickel - Molybdenum - Chromium Alloy Castings.'
A.S.T.M. Designation B332-58T pp. 675-8.

Covering corrosion-resistant and heat-resistant alloy materials in the form of sand, centrifugal and investment castings. The chemical compositions specified are as follows:

Nickel-Molybdenum Alloy

Chromium 1·00 max., iron 4·00–6·00, carbon 0·12 max., silicon 1·00 max., cobalt 2·50 max., manganese 1·00 max., vanadium 0·20–0·60, molybdenum 26·00–30·00, phosphorus 0·040 max., sulphur 0·030 max., per cent., nickel remainder.

Nickel-Molybdenum-Chromium Alloy

Chromium 15·50–17·50, tungsten 3·75–5·25, iron 4·50–7·50, carbon 0·12 max., silicon 1·00 max., cobalt 2·50 max., manganese 1·00 max., vanadium 0·20–0·40, molybdenum 16·00–18·00, phosphorus 0·040 max., sulphur 0·030 max., per cent., nickel remainder.

B. Standards in which Revisions have recently been made.

'Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock.'
A.S.T.M. Designation B111-58 pp. 172-7.

The specification covers tubing of 70-30, 80-20 and 90-10 copper-nickel alloys, various brasses and bronzes, and arsenical copper.

'Copper-Nickel-Zinc Alloy (Nickel Silver) Rod and Bar.'
A.S.T.M. Designation B151-58 pp. 270-2.

Covering nickel silver rod and bar in six alloys of the nickel silver type, containing 9–11, 11–13 or 16·5–19·5 per cent. of nickel, with varying amounts of copper and zinc.

'Copper-Alloy Condenser Tube Plates.'
A.S.T.M. Designation B171-58 pp. 284-8.

Covering rolled plates of seven copper alloys, for use as tube plates in surface condensers and heat exchangers. The types represented are leaded Muntz metal, naval brass, Admiralty metal, 70-30 and 90-10 copper-nickel alloy, and two grades of aluminium bronze.

Standard Hardness Conversion Tables

See abstract on p. 37.

Diffusion in the Solid State

See abstract on p. 37.

Cupro-Nickel Coinage in Spain

'A Mint of Money at Barcelona.'
The Times, 1958, Nov. 26, p. 9.

In a report of the first Ibero-American numismatic exhibition, held in November in Barcelona, attention is directed to the fact that the exhibition coincided with the issue of cupro-nickel coins of 5-, 25- and 50-pesetas denominations, which will gradually replace the notes at present in circulation.

Applications of High-Nickel Alloys in the Petroleum Industry

See abstract on p. 58.

NICKEL-IRON ALLOYS

Non-Linear Magnetics and Magnetic Amplifiers: Third Special Technical Conference

AMER. INST. ELECTRICAL ENGINEERS: 'Special Technical Conference on Non-Linear Magnetics and Magnetic Amplifiers: Proceedings.'

Published by the Institute, 1958; 482 pp.

The Conference, which was held at Los Angeles in August 1958, was sponsored jointly by the American

Institute of Electrical Engineers, the Institute of Radio Engineers and the Instrument Society of America.

The papers contributed were presented in sections classified as shown below.

Tutorial
Applications of Magnetic Amplifiers and Production Methods
Computer Magnetics
Application of Non-linear Magnetics
New Horizons

In addition to the general interest of much of the subject matter in relation to nickel and nickel alloys, several of the thirty papers have a specific bearing on nickel-containing materials. The scope of these papers is indicated in the following abstracts.

R. C. BARKER: 'Magnetization in Tape-Wound Cores', pp. 1–46.

The paper is 'tutorial', written with the aim of collating the available data and 'summarizing the state of knowledge on soft magnetic materials, in particular, as used in commercially available tape-wound cores. The summary is written from the electrical-engineering standpoint, and the language is purposely non-mathematical. The intent is to give a qualitative description of the physical and electrical properties of magnetic tapes in terms of simple experiments that can be performed in most laboratories without excessive special equipment.'

The first part of the paper, which outlines basic theory in terms of magnetization in single crystals, is applicable to magnetic materials in general. Sections are given on theory of ferromagnetic domains and the molecular field, energy of magnetization, and magnetization in a single cylindrical shell. The last section serves as an introduction to a summary of the basic principles relating to magnetization in cores of more complex fabrication.

The main portion of the review is devoted to discussion of the metallurgical and magnetic properties of significance in the production and application of tape-wound toroidal cores. The principles outlined are considered in connexion with 50–50 and 80–20 nickel-iron alloys and 97–3 iron-silicon alloys. They are, however, of general applicability to many other materials of different properties.

A résumé is given of the rolling and annealing procedures employed to achieve optimum grain size and orientation, and structures typical of those obtained are described with the aid of photomicrographs of three tapes of commercial grade: the iron-silicon alloy 'Magnesil', the nickel-iron-molybdenum alloy 'HyMu-80' and the nickel-iron alloy 'Orthonol'. Subsequent sections relate to magnetization phenomena in commercial core materials, and, in this connexion, discussion is limited mainly to the modifications necessitated in the basic principles (described in the first part of the review) by reason of the polycrystalline structure of the alloys used. Details are given of domain walls, domain rotation and domain nucleation in polycrystalline tape. Flux reversal in tape-wound cores is outlined, and the fundamental mechanism involved is illustrated by reference to experiments carried out mainly on nickel-iron cores. In a further section the author discusses the influence of power frequency and sinusoidal-voltage and sinusoidal-current excitation, on the shape of the hysteresis loop, and considers the behaviour of minor loops exhibited by rectangular-loop materials. This section also includes reference to the relation of the magnetic characteristics of tape-wound cores to circuit design.

The review ends with a brief account of high-speed flux reversal in tape-wound cores.

R. E. BALL: 'The Effects of Core Variations on Production Quantities of Magnetic Amplifiers', pp. 87–93.

The fundamental parameters of the core are factors which ultimately govern the performance of a magnetic amplifier. The author indicates how each parameter affects the behaviour of magnetic amplifiers of the self-saturating type, and outlines a method by which, using a sine current core analyzer, core characteristics may be measured and specified in a way more directly applicable to the needs of the magnetic-amplifier engineer. Use of such measurements has permitted maintenance of an overall production yield of more than 90 per cent. for some 30 different magnetic amplifiers, in production quantities of 500 or more.

Reference is made to measurements conducted on the nickel-iron-molybdenum alloy 'HyMu-80', 4–79 molybdenum-'Permalloy', and the iron-nickel alloys 'Orthonol' and 'Deltamax'. Among the data presented is a distribution curve of ΔH for a group of nickel-iron cores which gave an exceptionally poor yield in connexion with a particular design involving somewhat special gain requirements: from the data obtained it was ascertained whether these requirements were realistic, one of the extreme limits for gain requirements of the magnetic amplifier was determined, and the yield was eventually increased to more than 95 per cent.

J. E. WOLF: 'Influence of Excitation Frequency and Waveform on Squareness Ratio of Toroidal Tape Cores', pp. 94–112.

The paper gives the results of an investigation of the variation, with excitation frequency and excitation wave-shape, of the Br/Bm ratios of toroidal tape core materials of the non-rectangular-loop variety. Differences of up to 34 per cent. were found to exist between ratios obtained from symmetrical sine current loops and those obtained by the half sine wave-pulsing method. Br/Bm ratios were nearly doubled as a result of changes in frequency ranging from d.c. to 3600 c.p.s.

Details are given of a method of pulse testing which gives ratios showing close agreement with those derived from sine current hysteresis loops. Data are included on 'Supermalloy', 'Permalloy' and 'Deltamax'.

A. H. BOBECK and R. S. TITLE: 'The 'Twistor'—Its Use in a Memory Array', pp. 234–41.

The recently discovered 'twistor' offers advantages, both in economy and flexibility of design, over the ferrite cores or sheets conventionally used for coincident-current memory arrays. Use of the 'twistor' enables information to be inserted into a magnetic wire in the form of a polarized helical magnetization. Because the lines of flux wrap the wire, reversing the flux induces a voltage signal across the ends: the 'twistor' wire therefore serves not only as the storage element but also as the sensing means, the available signal of which is increased by the fact that the lines of flux wrap the wire several times.

The first part of the paper is devoted to a review of the 'twistor' principle and a discussion of its use as a magnetic storage element. The final section outlines the construction and operation of a 20×20 memory array in which 4–79 molybdenum-'Permalloy' is used as the magnetic material.

D. W. MOORE: 'The Preparation of Magnetic Films by High-Vacuum Evaporation', pp. 278–95.

The production of magnetic films by vacuum evaporation is a technique which is assuming increasing importance in the field of digital computers: a correctly prepared film with a square hysteresis loop of suitable geometry has, for example, proved ideal for use in such applications as memory elements.

The present paper falls into three sections. In the first the author describes the procedures employed in the production of magnetic films by electron-bombardment evaporation. The second section deals with apparatus and techniques used to determine hysteresis curves, coercive forces and film thickness. The final section reviews, by way of example, the evaporation techniques used to obtain, and properties typical of, iron, iron-nickel, iron-silicon and iron-cobalt magnetic films.

Much of the author's work was carried out on cores containing 80 per cent. nickel and 20 per cent. iron. Some of the advantages of this alloy, in relation to its use for magnetic memory units, are illustrated by reference to curves relating to the Curie point, coercive force and strain sensitivity. Coercive force reaches a minimum in the vicinity of 80 per cent. nickel. Strain sensitivity, which is important in connexion with possible variations between the coefficients of expansion of film and basis material, reaches zero at about the same content. The initial and maximum permeability exhibited by the alloy with 80 per cent. nickel is greatly increased by heat-treatment. (Highest maximum permeability, *per se*, is found in alloys containing 68 per cent. nickel, but values for other desirable properties are lower.)

Typical hysteresis loops obtained for iron, iron-nickel and iron-silicon films are presented. Coercive forces of less than 0·3 oersted and saturation values of approximately 3000 gauss were exhibited by 80–20 nickel-iron films approximately 200 Å thick.

R. L. CONGER: 'Thin-Film Magnetization-Reversal Studies', pp. 444–53.

The author discusses the findings of research into the characteristics of evaporated magnetic films, carried out with the initial aim of devising faster and less expensive coincident-current memories. The magnetic film has, however, also provided a useful means for fundamental study of the dynamics of magnetization reversal, and the major portion of the paper is concerned with discussion of this subject.

The author advances a theory which interprets reversal in evaporated films at low fields in terms of domain-wall motion, with the number of moving walls determined by a simple statistical model which allows for the random nucleation of walls as the reversing field increases. The number of walls increases with increasing field until, at the anistropy field, the film is completely filled with walls. The process then changes over to one of domain rotation, which is the factor governing magnetization reversal at high fields.

Investigation of the suitability of thin films for use as memory elements showed that the de-magnetizing field of an 80–20 nickel-iron film 1 cm. square and 2×10^{-5} cm. thick is so small that the film, with an open flux path, can have a rectangular hysteresis loop. A magnetic-film memory can thus be built simply by sandwiching together a rectangular array of evaporated squares and printed-circuit sheets. The method is illustrated.

R. E. JACKSON: 'The Use of Magnetic Amplifiers at High Frequencies', pp. 454–64.

Problems encountered in magnetic amplifiers operating at high frequencies are discussed, and design equations are presented by means of which an amplifier, for use at a clock rate of 500 KC, was constructed so as to ensure minimum deviation of characteristics over large production quantities, Finally the gain of the units is considered in relation to determination of optimum operating conditions. Using a 4-79 molybdenum-'Permalloy' core, the maximum gain was 11·2 and 10 at outputs of, respectively, 95 and 70 ma.

Influence of Tensile Stress on Austenite→Martensite Transformation in Iron-Nickel Single Crystals

A. KOCHENDÖRFER and G. OTTO: 'Influence of Tensile Stress on Austenite-Martensite Transformation in Iron-Nickel Single Crystals.'

Archiv f.d. Eisenhüttenwesen, 1958, vol. 29, Nov., pp. 709–14.

The investigation described forms a continuation of the experiments, reported previously by KOCHENDÖRFER and MÜLLER (*ibid.*, 1955, vol. 26, pp. 291–8), which were carried out on iron-nickel single crystals in an attempt to minimize the number of orientations assumed by martensite. During the austenite→martensite transformation the crystals were subjected to a tensile stress which, it was hoped, would, in the initial stages of transformation, induce a preferred

orientation in the martensite. Simultaneously with the applied stress, a temperature gradient was maintained along the length of the crystals, with the aim of inducing transformation in the direction of the gradient and so, by nucleation, maintaining the preferred orientation throughout. Cooling the crystals to slightly below the M_s point gave rise to only about four orientations in the martensite, the number of orientations increasing with fall in temperature until the preferred orientations became indistinguishable. The stress applied was approximately 75 per cent. of the room-temperature yield point, and the temperature gradient ranged from room temperature at one end of the specimen to the temperature of liquid air at the other. The yield point of the specimen increases, however, with fall in temperature, due partly to the effect of temperature *per se*, partly to the increase in the amount of martensite present, and it was anticipated that a proportionate increase in the applied stress would increase the amount of uniformly oriented martensite. Moreover, use of a gradient involving only a small temperature range was expected to prove more beneficial, enabling the uniformly oriented needles to extend along the crystal without interruption by needles of different orientations formed by extremes of temperature. The present paper gives results of experiments made under these new conditions.

A vacuum-melted iron-nickel alloy of high purity, containing 30 per cent. of nickel, and with an M_s point at about $-30°C.$, was selected for investigation. To minimize the effects of residual stress, tensile specimens machined from the single austenitic crystal were annealed at 1200°C. after electropolishing. The temperature gradient maintained along the length of the crystal ranged from 10° to 20°C.; the stress applied was maintained at 75 per cent. of the actual yield point at any one moment during slow cooling to the temperature of liquid air.

A structure consisting of large areas of predominantly uniformly oriented needles of martensite, alternating with smaller regions of various other orientations, was formed as a result of cooling to about $-45°C.$ The only effect of further cooling was to produce additional needles of similar or different orientation, which filled the regions between the initial needles. The authors conclude that the applied stress is the main factor influencing the orientation of the needles and that a sufficiently high tensile stress will achieve long needles with a small number of orientations. Transformation effected with no stress, or with an insufficiently high stress, has no effect on the number and frequency of the orientations formed by martensite at low temperatures. The temperature gradient determines the distribution of the martensite along the crystal.

See also following abstract.

A. KOCHENDÖRFER and G. OTTO: 'X-Ray Investigation of the Austenite-Martensite Transformation in Iron-Nickel Crystals under Tensile Stress.'
Ibid., Dec., pp. 775-84.

The paper is the sequel to that referred to in the previous abstract. In it are recorded the results of X-ray determinations of the four or five preferred orientations exhibited by the martensite as a result of slow cooling under the experimental conditions described.

CAST IRON

Chill-Casting of 'Ni-Hard' Rolls

J. D. KELLER and N. R. ARANT: 'The Air Gap in Permanent-Mold Casting.'
Blast Furnace and Steel Plant, 1958, vol. 46, Sept., pp. 957-65.

The depth of hardening achievable in chill-cast iron rolls of specific composition and diameter is limited, and hardness values are much inferior to those obtained in components of thin section. These limitations are attributable mainly to the 'air' gap (i.e., the gas-filled gap) which forms between the mould and the solidified surface of the casting, and acts in some degree as an insulator between the metal and the chiller. The investigation described was carried out, under the aegis of the Roll Manufacturers' Institute, to supplement the sparse information available on the formation and behaviour of the gap: in particular, the experiments comprised the first attempt to obtain a reasonably accurate determination of the width of the gap and its growth, with time, during the chill-casting of rolls.

Three production sizes of alloy-cast-iron rolls were selected for investigation. The width and growth of the gap were determined by analysis of heat conduction and heat storage in the chiller and of temperature changes in the casting itself. The necessary measurements were made by means of thermocouples embedded at various depths in the mould and the body of the roll. Growth of the air gap was studied only in the first 15 minutes after pouring, since the chilled shell had finished forming within that time. The data obtained are presented in graphical form. Details are given of the calculations involved and the casting procedures used.

Variation of the width of the gap as a function of time and the diameter of the roll is discussed. The curves presented show that the air gap does not occur as a sudden or 'catastrophic' phenomenon, but that it exists initially and then increases, at first rapidly, and then more slowly along a smooth curve. No definite conclusion is reached on the influence, on the width of the gap, of the diameter of the roll, but the results indicate that diameter is probably of less significance in this respect than the effects of 'swirl' of the molten metal entering the mould, the thickness of the layer of blacking previously applied to the inside of the mould, and eccentric positioning of the casting in the mould.

It is emphasized that while the conclusions reached are of specific interest only in relation to rolls produced in 'Ni-Hard' cast iron, many of the findings are generally applicable to other kinds of materials and to chill- or permanent-mould casting (e.g., the solidification of steel ingots in their moulds).

CONSTRUCTIONAL STEELS

Forms of Nickel available as Alloying Additions for Steels

'Nickel in Special Steels.'
Metal Bull., Ferro-Alloys Special Issue, 1958, Nov., pp. 91 and 94.

Although in recent years demand for nickel has exceeded supply, the metal is now freely available, and extensive development of virgin deposits has ensured that it will remain so. Full advantage can thus be taken of the beneficial effects which nickel confers on the physical mechanical and heat-treatment characteristics of steels. In this connexion the article gives details of the forms in which the metal is available for addition to steels, and briefly summarizes the addition methods used. The information relates to nickel pellets, electrolytic nickel, nickel-oxide sinter, 'F' nickel and nickel-magnesium alloys.

Effect of Shot-Peening prior to Chromium Plating, on Fatigue Strength of Chromium-Nickel-Molybdenum Steel

B. COHEN: 'Effect of Shot-Peening prior to Chromium Plating, on the Fatigue Strength of High-Strength Steel.'
45th Ann. Tech. Proc., Amer. Electroplaters' Soc., 1958, pp. 33–6; disc., pp. 159–60.

The high residual tensile stresses present in electrodeposited chromium coatings have been found drastically to reduce the fatigue strength of the plated steel: the deterioration becomes more severe with increase in the tensile strength or hardness of the steel. The properties which contribute to the excellent abrasion-resistance of chromium coatings are thus precisely those which have so deleterious an effect on fatigue strength, and the range of applications for which the coatings may be used is therefore severely limited. Treatment of the basis metal prior to plating, to relieve or counteract the stresses in the chromium, has not proved entirely satisfactory, since improvement in one property has generally been accompanied by adverse changes in others. Previous investigators had shown, however, that, in the case of steels with tensile strengths in the region of 180,000 p.s.i. (80·5 t.s.i.; 126·5 kg./mm.^{2}), the residual compressive stresses produced on the surface of the steel by shot-peening before plating would counteract the tensile stresses of the chromium deposit and minimize its deleterious effects. The work described was carried out with the aim of (1) determining the effects of similar treatment on the fatigue strength of chromium-plated high-tensile and ultra-high-tensile steel, and (2) establishing whether any improvement so effected would be maintained throughout subsequent baking for correction of hydrogen embrittlement.

The steel used as basis material was of the following composition: carbon 0·39, silicon 0·25, manganese 0·73, sulphur 0·010, phosphorus 0·012, nickel 1·74, chromium 0·81, molybdenum 0·24, per cent. Fatigue specimens were heat-treated to two levels of strength (220,000 and 280,000 p.s.i.; 98 and 125 t.s.i.; 155·5 and 197 kg./mm.^{2}), machined and polished. One third were used as controls; the others were shot-peened and chromium-plated. Half of the chromium-plated specimens from each group were finally baked for 3 hours at 375°F. (190°C.). All the specimens were then fatigue-tested.

Shot-peening was found to have a highly beneficial effect on fatigue strength, a finding which is reflected in an amendment to *Federal Specification QQ-C-320*, 'Chromium, Electrodeposited', specifying such treatment prior to plating, in all applications involving conditions liable to cause fatigue.

The author summarizes his conclusions as follows: The chromium-plated high-strength steel specimens used in this investigation showed only slight fall in fatigue strength as a result of plating, by comparison with the drastic fall typical of specimens which have not been shot-peened prior to plating.

Shot-peening of steels of medium strength not only counteracts the harmful effects of chromium-plating, but actually results in increase in fatigue strength of the coated steel, as compared with the bare polished material. Shot-peened chromium-plated specimens which had been baked after plating showed an endurance limit 18 per cent. higher than that of the unplated material: specimens not baked after shot-peening and plating had an endurance limit 12 per cent. higher than that of the uncoated steel.

When subjected to shot-peening of a given intensity the higher-strength steel shows greater resistance to plastic deformation than the medium-strength steel and, as a result, the cold-working effect is not so marked on the high-strength steel as on that of lower strength. A thinner layer of residual compressive strength is therefore induced at the surface, an effect which accounts for the difference in degree of improvement obtained in the two steels from shot-peening before chromium plating. It is believed that a higher intensity of shot-peening, which would result in greater plastic deformation of the high-strength specimens, would probably increase the effectiveness of the treatment: there is possibly a correlation between optimum intensity of shot-peening and the tensile-strength level of the steel.

Baking chromium-plated shot-peened specimens at 375°F. (190°C.) slightly increased fatigue strength.

Machining and Assembly of the 'Rotodyne' Rotor Blade

''Rotodyne' Rotor-Blade. Part I. Heat-Treatment and Machining Operations on the Main Spar. Part II. Forming Stainless-Steel Ribs and Skin: Aerofoil Assembly.'
Aircraft Production, 1958, vol. 20, Nov., pp. 437–42; Dec., pp. 483–8.

The 'Rotodyne' vertical-take-off aircraft developed by the Fairey Aviation Company, Ltd., has been

the subject of much interest since its appearance at the 1958 Farnborough Air Show. For vertical take-off, use is made of a power-driven rotor, while for forward flight the rotor autorotates, and propulsion is supplied by twin airscrew turbines mounted on a short-span fixed wing of conventional two-spar design. Lift in forward flight is shared between the wing and the autorotating rotor.

Each of the four 35-ft. (10·5-m.) blades of the rotor is driven by a tip-jet unit and is of symmetrical aerofoil section. Structurally, the blade is of conventional two-spar wing structure, with a main leading-edge spar of high-tensile steel, a rear spar of channel section, and inter-spar ribs. The last two components are fabricated from chromium-nickel stainless-steel sheet: the material for the rear spar and the outer skin is to D.T.D. Specification 166 (carbon $\geqslant 0{\cdot}2$, nickel $\leqslant 6$, chromium $\leqslant 12$ per cent.), which provides for a 0·1 per cent. proof stress of 40–50 tons per sq. in. (63–79 kg./mm.²); steel for the inter-spar ribs is to D.T.D. Specification 171, which stipulates a similar composition, but a 0·1 per cent. proof stress of not less than 15 tons per sq. in. (23·5 kg./mm.²). The rear spar is made from two press-formed L-section strips riveted together to give a channel section; the ribs are attached to the outer skin by flush 'Monel' rivets. The main leading-edge spar of the rotor blade is machined from a single billet of low-alloy chromium-molybdenum-vanadium high-tensile steel.

The leading-edge spar is reduced, in its final form, to very thin sections, and, in addition to overcoming the problems inherent in the machining of a high-tensile steel, means had to be found to maintain the straightness of the spar throughout machining and heat-treatment. These and other difficulties encountered in fabrication of the spar, together with the techniques and equipment evolved to overcome them, form the subject matter of the first part of the article. The second instalment describes the tooling for the stainless-steel ribs and the aerofoil skin, and gives details of the procedure used to assemble the blade.

HEAT- AND CORROSION-RESISTING MATERIALS

Changes in the Structure of Austenitic Steels during Creep Testing

W. KOCH, A. SCHRADER, A. KRISCH and H. ROHDE: 'Changes in the Structure of Austenitic Steels during Creep Testing.'

Stahl und Eisen, 1958, vol. 78, Sept. 4, pp. 1251–62.

The high-temperature behaviour of austenitic chromium-nickel steels, stabilized and unstabilized, with and without molybdenum or other minor additions, has been the subject of much research. Structural changes resulting from high-temperature exposure have, for example, been reported by BUNGARDT and LENNARTZ (see abstracts in *Nickel Bulletin*, 1956, vol. 29, No. 8-9, p. 172; 1958, vol. 31, No. 9-10, p. 271). Little attention has, however, been paid to one factor of importance in relation to the creep behaviour of the steels, i.e., the influence of stress or creep on the progress of precipitation and on changes in the nature of the precipitates. A comprehensive series of creep tests carried out on a wide range of austenitic steels presented the authors with the opportunity of studying these relationships. Their findings are recorded in the present paper.

Four steels were selected for study, two stabilized with titanium, the others with niobium+tantalum. Their compositions and the heat-treatments used for the test materials are given in Table I on p. 50. The steels were exposed at 600°, 650° and, in some cases, also at 700°C., for times between 1 and 50,000 hours and at stresses in the range 0–40 kg./cm.² (0–25·5 t.s.i.). Some of the specimens were tested to rupture, the others for specific exposure times. Determination and structural identification of the precipitates present, and study of microstructural changes as a function of time and applied stress, were effected by X-ray and electron microscopy, and by examination and micro-analysis of electrolytically-isolated precipitate particles. The findings, which are summarized below, are illustrated by numerous photomicrographs.

Composition, quantity and structure of the precipitates was found to be determined by time and temperature of testing and the stress used. Precipitation proceeded similarly in all four steels. In the condition prior to testing the steels contained only the monocarbide, MC, of the stabilizing element. Creep-testing for a relatively short period resulted in precipitation, particularly within the grains, of very fine particles of the MC carbide and, preferentially at the grain boundaries, of the chromium-rich cubic $M_{23}C_6$ phase. There were indications that these phases formed by way of intermediate structures observed at the grain boundaries. In all the steels the $M_{23}C_6$ carbide broke down as a result of longer heating and was replaced by sigma phase and by further amounts of MC carbide: unidentified transition structures also were observed during this reaction. In all cases, rupture occurred at an earlier phase of the precipitation reaction as the stress was increased. The only fundamental difference between the individual steels was in the time required for the various stages of precipitation and in the amount of the phases formed. Precipitation occurred more rapidly in the titanium- than in the niobium-stabilized steels.

Precipitation Behaviour of Nickel-Chromium-Molybdenum-Iron Alloy

M. PRÖTZL: 'Precipitation Behaviour of an Alloy with 15% Cr, 16%Mo and 55% Ni.'

Archiv f.d. Eisenhüttenwesen, 1958, vol. 29, Dec., pp. 745–54; disc., pp. 754–6.

Although materials containing about 15 per cent.

Table I

Chemical Composition and Heat-Treatment of Creep-Tested Steels

(*See abstract on p. 49*)

Steel	C %	Si %	Mn %	S %	P %	Ni %	Cr %	Mo %	Nb+Ta %	Ti %	N %	Heat-Treatment %
1	0·060	0·48	0·31	0·030	0·019	10·75	16·56	0·06	—	0·42	0·013	1 hr. 1050°C./water-quenched
2	0·081	0·50	0·67	0·009	0·020	10·05	18·20	0·22	—	0·64	0·012	20 min. 1080°C./water-quenched
3	0·056	0·40	1·24	0·018	0·015	13·55	16·95	0·18	0·69	0·03	0·019	20 min. 1100°C./air-cooled
4	0·064	0·59	1·41	0·008	0·022	12·05	17·30	2·22	0·64	0·06	0·023	20 min. 1100°C./air-cooled

chromium, 16 per cent. molybdenum and 55 per cent. of nickel have achieved considerable technical importance, the fundamental data available, particularly on their precipitation-hardening behaviour, are extremely sparse, and relevant literature (which is discussed by the author) pertains mainly to binary and quaternary systems with a high iron content.

The present paper gives the results of experiments carried out to investigate, as a function of time and temperature, the form and distribution of the precipitates occurring in an alloy of the following composition: nickel 54·95, molybdenum 15·82, chromium 14·8, cobalt 1·75, silicon 0·61, manganese 0·47, vanadium 0·34, carbon 0·08, phosphorus 0·03, sulphur 0·004, per cent., remainder iron.

The alloy was hot-worked to bar, which was then heated to 1230°C., water-quenched and finally machined to the desired test piece. Solution and precipitation reactions were studied by microstructural examination of specimens heated for various times at various temperatures and finally water-quenched. Photomicrographs of structures typical of those resulting from annealing for various times at 1150°, 900°, 850° and 825°C. are presented and discussed.

The information obtained is summarized in a time/temperature/precipitation diagram illustrating the regions in which five characteristic structures are produced. Use of this diagram enabled the author to prepare specimens of a pre-determined structure with which, in further experiments, investigation was made of (1) the influence, on the lattice parameter, of the quenching temperature and the amount of precipitate present, and (2) the effects, on the initial structure, of subsequent heat-treatment intended to produce an entirely different condition of precipitation. Other parts of the investigation involved identification of the phases present and determination of the corrosion-resistance (to 1 and 40 per cent. hydrochloric acid, at 80°C. and boiling point) and of the notch-sensitivity of specimens heat-treated to each of the five conditions of precipitation.

Metallographic examination showed that sigma phase and the carbides M_6C, $M_{23}C_6$ and Mo_2C were present in an austenitic-type matrix. The M_6C phase occurred in all the conditions of heat-treatment and was stable up to the melting point; the $M_{23}C_6$ precipitated simultaneously with the sigma phase and preferentially around the M_6C carbide. The sigma phase precipitated at the grain boundaries in a chain-like structure, and, under favourable conditions, needles of sigma formed within the grains. Precipitation was found to have a deleterious effect on intercrystalline cohesion, but the tendency of the sigma phase to form intragranular needles with increase in time and temperature and decrease in surface energy nullified the embrittling effect exhibited by the phase at lower temperatures. It is concluded that satisfactory corrosion-resistance and notch-sensitivity are dependent either on (1) taking the deleterious grain-boundary precipitates into solution by quenching from 1250°C., or (2) transforming the shape and location of the precipitate by long-time annealing (e.g., for 240 hours at 1100°C.).

The practical conclusions drawn by the author from his findings are summarized below.

Cracking during hot working can be prevented by long-time annealing (at the temperature indicated) to break up the deleterious grain-boundary precipitates. Use of a higher temperature would result in solution of precipitates already present in beneficial form; heating at a lower temperature, or insufficiently rapid cooling from the quenching temperature, would induce further grain-boundary precipitation. Where the degree of deformation is small enough to require no preliminary annealing, and the component is of suitable dimensions, quenching from a temperature above 1200°C. is recommended as the more economical way of preventing embrittlement. Stress-relief treatment would involve heating at the temperature from which the alloy was quenched or at that used for long-time annealing.

Scaling Characteristics of Nickel-Chromium Alloys

C. A. BARRETT, E. B. EVANS and W. M. BALDWIN: 'Thermodynamics and Kinetics of Metals and Alloys. High-Temperature Scaling of Ni-Cr, Fe-Cr, Cu-Cr and Cu-Mn Alloys.'

U.S. Office of Ordnance Research, Tech. Report, Dec. 1955 (recently declassified); 30 pp. + figs.

The report is the third in a series of three summarizing the results of investigation of the scaling behaviour, in air at high temperatures, of alloys of the following systems: nickel-manganese, cobalt-chromium, nickel-chromium, iron-chromium, copper-chromium and copper-manganese. Data derived from study of cobalt-chromium alloys were recorded in the second of the reports, dated November 1955. Reference was made to the salient findings relevant to the nickel-manganese alloys in *Nickel Bulletin*, 1956, vol. 29, No. 8-9, p. 170. This last report is in four sections, each presenting the results of experiments on one of the remaining alloy systems studied. Each section is therefore an entity in itself (though comparisons are drawn among the various systems), and all are similar in format: information available in the literature is summarized, the materials used are described, details are given of a kinetic study of the scaling process, and the results of metallographic examination of the scales and subscales formed are discussed. The report is supported by a *bibliography* of 56 items.

The scope of the test programme, insofar as it relates to the nickel-chromium alloys, is indicated below.

Three alloys were selected for study: two were prepared by vacuum melting from high-purity materials, and contained, respectively, 36·3 and 68·2 wt. per cent. of chromium; the third was of commercial 80-20 nickel-chromium type. The alloys were exposed in air for various times at temperatures in the range 700°-1100°C.

In general, all three alloys were found to scale in accordance with the parabolic rate law: there was, however, some deviation to a lower rate after long times of scaling. Scaling behaviour was similar to that observed on cobalt-chromium alloys, and the patterns obtained were, in general, the reverse of those for the nickel-manganese alloys. Instead of being straight lines from the original alloy composition to the oxygen corner of the diagram, the scaling patterns veer first to the chromium-rich and then to the nickel-rich side, implying that the nickel (or cobalt) ion diffuses outwardly at a much higher rate than the chromium ion. When the chromium content of the original alloy was high (above a critical concentration of about 25 per cent.) the scale consisted exclusively of Cr_2O_3 and the change in direction of the path to the nickel-rich side did not occur.

At a given temperature, the scaling rate of the alloys first rose with low chromium additions and then dropped precipitously with increase in the chromium content, to reach a minimum at about 20-25 per cent. chromium. Above that level the scaling rate increased with increasing chromium content, to reach a maximum at pure chromium. Optimum resistance to scaling was therefore associated with a chromium content of 20-25 per cent., i.e., the minimum necessary for exclusive formation of Cr_2O_3.

Thermal-Fatigue and Related Properties of High-Temperature Alloys

F. J. CLAUSS and J. W. FREEMAN: 'Thermal Fatigue of Ductile Materials.

I. Effect of Variations in the Temperature Cycle on the Thermal-Fatigue Life of 'S-816' and 'Inconel 550'.

II. Effect of Cyclic Thermal Stressing on the Stress-Rupture Life and Ductility of 'S-816' and 'Inconel 550'.'

Nat. Advisory Committee for Aeronautics, Tech. Notes 4160 (61 pp.) and 4165 (43 pp.), Sept. 1958.

Modern trends in various branches of high-temperature engineering are increasingly towards conditions involving not only higher service temperatures but also, in many cases, cyclic operations covering wide ranges of temperature. Research on thermal fatigue has, however, been centred mainly on brittle materials, and few data are available on the behaviour of ductile alloys under alternations of temperature. Moreover, where laboratory data have been available there has been little success in correlating them with service life of high-temperature components.

The National Advisory Committee for Aeronautics therefore undertook fundamental research, the results of which are presented in these two reports.

Technical Note 4160 reviews the literature on thermal fatigue, and presents results indicating the effects of various test conditions on the number of cycles to complete failure in the two materials named.

Technical Note 4165 reports a study of the changes in the stress-life and ductility of the materials after exposure to varying amounts and conditions of thermal fatigue, employing a number of cycles insufficient to cause fracture by thermal fatigue alone. Such data may be regarded as being helpful not only in relation to engineering design but also in clarification of the mechanism of thermal fatigue.

The two alloys tested were selected as typical of precipitation-hardening alloys used in turbojet engines. 'Inconel 550' is a nickel - chromium - base alloy containing titanium, aluminium and niobium: 'S-816' is a complex cobalt-nickel-chromium alloy containing molybdenum, tungsten, niobium and iron. Both are used mainly in service in the temperature range 1350°-1500°F. (730°-815°C.): both were tested in the condition in which they are employed in service.

The introduction to the first report contains (1) a survey of basic concepts of thermal stress, thermal shock and thermal fatigue and the effects resulting from the existence of such conditions; (2) a brief consideration of tests used for evaluation of the thermal-stress resistance of high-temperature materials; (3) a detailed survey of relevant literature. A *bibliography* of 21 items is appended to the report.

Equipment and procedure used in the authors' experimental work are described at length and well illustrated. The cycle through which the specimens

were heated and cooled was divided into four parts: (1) heating to T_{max}, (2) holding at T_{max}, (3) cooling to T_{min} and (4) holding at T_{min}. The maximum cycling temperature ranged from 1150° to 1600°F. (620°–870°C.): the minimum temperature was in most cases 200°F. (93°C.), but in a few cases was 500°F. (260°C.). The test cycle usually followed consisted in (1) heating in 30 seconds to T_{max}; holding for 15 seconds at T_{max}; cooling in 30 seconds to T_{min}; holding 15 seconds at T_{min}, before repetition of the cycle. In some tests the time of holding at T_{max} was increased to 60 seconds. The number of cycles to fracture, changes in hardness, and microstructural behaviour were studied as a function of the conditions of temperature cycling.

The main conclusions drawn from the results of the tests are as follows:

The number of cycles to failure was more sensitive to changes in the maximum temperature than to changes in difference of temperature.

Lengthening the time of exposure at high maximum cycle temperatures increased the number of cycles to failure, whereas the same increase in time at lower maximum cycle temperatures had the reverse effect.

The number of cycles to failure depends upon temperature and time effects, as well as on the thermal strains absorbed by plastic flow in the materials.

In view of the complex effects imposed by rapid alternations of temperature, it can be presumed that thermal-fatigue conditions could weaken a material so that it would fail in service from other loading stresses before failure would occur as a result of thermal fatigue alone. Conversely, strain- and/or precipitation-hardening occurring during constrained thermal cycling might strengthen a material. Further research was therefore carried out to study the changes in stress-rupture life and ductility of materials which had been exposed to varying amounts and conditions of thermal fatigue, but had not been tested to the point of obtaining fracture from that cause.

Equipment and procedure were essentially the same as in the earlier tests, i.e., tensile specimens of 'S-816' and 'Inconel 550' were alternately heated and cooled while constrained in a manner preventing free axial expansion and contraction, but instead of continuing the tests until the specimens fractured, thermal cycling was interrupted at an earlier stage and the specimens were metallographically examined or tested in stress-rupture, mainly at 1350°F. (730°C.) and, in a few cases, at 1500°F. (815°C.). In most of the cycling tests the maximum temperature was 1350°F. (730°C.) and the minimum 200°F. (93°C.): in other tests a T_{max} range of 1250°–1500°F. (675°–843°C.) was studied. A few specimens were first run for short periods in stress-rupture and were then failed by thermal fatigue.

The conclusions drawn from this series of tests are summarized below:

Exposure to thermal-fatigue conditions strengthened 'S-816' in stress-rupture, but weakened 'Inconel 550'. In specimens first exposed to stress-rupture conditions and afterwards run to failure by thermal fatigue, the thermal-fatigue life of 'S-816' was sharply reduced, whereas that of 'Inconel 550' showed a slight increase.

It is considered that these results can be interpreted by extending existing theories of mechanical fatigue and creep-rupture to thermal fatigue. In this extension, the thermal-fatigue process is divided into two stages: first, a stage of strain- and/or precipitation-hardening, during which ductility is reduced and strength is increased; second, a stage in which there is destruction of cohesive bonds and development and propagation of cracks. Structural changes are an important part of thermal-fatigue behaviour.

An appendix to this report contains a review of the literature on the mechanism of failure of metals, covering factors in the deformation of metals which may be significant in relation to the behaviour of materials during reversed deformation under cyclic temperatures. The following aspects are included: theory of low-temperature deformation, the Bauschinger effect, theories of high-temperature creep, mechanical fatigue, and thermal fatigue. A *bibliography* of 33 items, attached to the report, is concerned mainly with this review.

Fabrication of 'Nimonic' and Austenitic Stainless-Steel Sheet

H. ROBERTS: 'Fabrication of Modern Heat-Resisting Materials for the Gas-Turbine Industry.'
Welding and Metal Fabrication, 1958, vol. 26, Dec., pp. 435–42 and 454.

In the manufacture of gas turbines, the excellent heat- and corrosion-resisting characteristics of the 'Nimonic' nickel-chromium-base alloys and stainless steels of chromium-nickel-austenitic type have been exploited to advantage in many applications. The 'Nimonic' alloys have, since the development of the aircraft gas turbine, been the standard materials in Great Britain for rotor blades, and the suitability of these alloys, and of the stainless steels, for fabrication in the form of sheet, is exemplified by their use for such components as combustion and exhaust units. With the aim of assisting other fields of industry in which these alloys and steels, in the form of sheet, are now finding increasing application, the author summarizes in this article the experience acquired in fabricating materials for gas-turbine parts. Discussion is limited, in the case of the alloys, to 'Nimonic 75', '80A' and '90'; the austenitic steels are represented by those of 18-8, 23-11 and 25-20 types.

Data are tabulated on the physical and mechanical properties of each grade of material considered, and, since heat-treatment often influences the stage at which welding is carried out, some information is also given in this connexion. Short sections are included on pressing and deep drawing, spinning, and blanking, and surface de-oxidation prior to

welding. Fabrication of the alloys and steels by welding is dealt with in rather more detail: notes on the suitability of available welding processes serve as an introduction to a review of optimum procedures and conditions for use in welding the materials by the metal-arc-, argon-arc- and resistance-welding techniques. A section covering joining of the alloys and steels by riveting is followed by a brief general outline of precautions necessary in welding them to dissimilar materials.

Cast Alloys for High-Temperature Service

1. J. T. BROWN: 'Nicrotung.'
2. J. E. WILSON: 'DCM Alloy.'

Metal Progress, 1958, vol. 74, Nov., pp. 83–7.

In the major countries concerned with high-temperature engineering, fundamental research is now being actively carried out to supplement the more empirical approach necessitated by the urgency of wartime conditions. Current literature bears evidence of the variety of materials under study, and of the principles being adopted in efforts to produce alloys having improved properties, at continuously rising levels of service temperature and stress.

Among the avenues being explored is that of the cast alloys, and it is with two representative materials of that group that these articles are concerned.

(1.) The alloy 'Nicrotung', evolved by Westinghouse Electric Corporation, has been developed to meet the requirements for a material having a 100-hour stress-rupture strength of 20,000 p.s.i. (9 t.s.i.; 14 kg./mm.^{2}) at 1800°F. (980°C.) and capable also of being produced by large-scale methods already used by Westinghouse. Basic considerations in selection of the composition were the following:

A considerable background of experience in high-temperature alloys indicated nickel as the basis element, because it is the most practical high-melting-point element for service at 1800°F. (980°C.). Chromium was added as the element conferring oxidation-resistance on the nickel-base alloy, and tungsten as the hardening element (used to the limit of solid solubility in the basis alloy). Properties of the nickel base were enhanced by adding a small amount of cobalt, but not enough to exceed solid-solubility limits. Precipitation-hardening by dispersion of small particles of a second phase was accomplished by adding titanium and aluminium, and finally, additions of small, but critical, percentages of boron, zirconium and carbon were made, to effect still further improvement in the alloy.

A nominal composition is given as chromium 12, cobalt 10, tungsten 8, aluminium 4, titanium 4, carbon 0·10, boron 0·05, zirconium 0·05, per cent., balance nickel.

Experiments with seven different atmospheres for melting and casting have indicated that both vacuum- and argon-melting and -casting result in good properties. When melted or cast in air, the alloy displayed inferior and less predictable properties. Tests to determine the influence of the minor elements present in the alloy have led to the conclusion that without at least some minimum level of carbon, boron or zirconium it is impossible to make sound castings. Carbon appears to be the best element for increasing the strength, followed by boron and zirconium. The combination of any two of these elements is better than any one used alone, and best results are achieved by using all three together.

The microstructure of the alloy consists of a solid-solution matrix, with a small amount of teardrop-shaped second phase, which usually occurs at grain or sub-grain boundaries. Typical photomicrographs are shown.

Charts show the mechanical properties of 'Nicrotung' at various temperatures up to 1800°F. (980°C.), also stress-rupture times at various temperatures over the range 1500°–1900°F. (815°–1035°C.).

(2.) 'DCM Alloy', developed by General Electric Company, Cincinnati, was evolved as the result of a statistically planned study of 27 compositions involving variation of three elements, each studied at three levels. It is stated that the alloy selected from these tests as optimum gave an average stress-rupture life of 525 hours at 15,000 p.s.i. (6·7 t.s.i.; 10·5 kg./mm.^{2}) and 1800°F. (980°C.).

(The designation 'DCM' derives from the initials of the three individuals mainly concerned in its development: E. L. Dunn, M. E. Cieslicki, and J. B. Moore.)

The preferred composition range of 'DCM Alloy' is as follows: carbon 0·08 max., manganese 0·10 max., silicon 0·15, sulphur 0·015 max., chromium 14·0–16·0, titanium 3·35–3·65, boron 0·070–0·090, aluminium 4·4–4·8, molybdenum 4·5–6·0, iron 4·0–6·0, copper 0·10 max., per cent., balance nickel. Heats up to 1000 lb. (2200 kg.) have been produced by vacuum-induction melting: blades are produced from the master melt by investment casting *in vacuo*. Research during the development of the alloy has covered study of many variables, including method of addition of reactive elements, partial addition in the master melt with final addition in re-melts, time allowed for various stages of melting, effect of the configuration of the cast, effect of vacuum re-melting, and effect of grain size. The last-named variable proved to be of major significance: data recorded in this article show that as the average grain diameter increases from 0·0035 (0·0875 mm.) to about 0·200 in. (5 mm.) rupture life of the alloy, measured at 1800°F. (980°C.) and 15,000 p.s.i. (6·7 t.s.i.; 10·5 kg./mm.^{2}), increases from 70 to 500 hours.

The author makes a comparison of the rupture life of 'DCM Alloy', over a range of temperatures and stresses, with values characteristic of other cast high-temperature alloys. Further information given includes tensile properties at elevated temperatures and data on density, coefficient of thermal expansion and thermal conductivity. Attention is directed to satisfactory retention of fatigue strength at high temperatures.

Heat-treatment procedure used on the specimens of 'DCM Alloy' from which the reported values are taken was as follows:

(1) Solution treatment at 2100°F. (1150°C.) for one hour; air-cooling.
(2) Ageing at 1950°F. (1065°C.) for two hours; air-cooling.
(3) Ageing at 1550°F. (845°C.) for four hours; air-cooling.

Use of 'Inconel X' in Research Aircraft for Space Flight

'Manned Missile: 'X-15.' '

Machine Design, 1958, vol. 30, Oct. 30, p. 12.

This short article gives details of the dimensions, construction and expected performance of the research aircraft 'X-15', which is due to make its first flight this year. The aircraft, which was developed by North American Aviation Company, will be air-launched to reach speeds of more than 3,600 miles per hour, and altitudes of 150 to 300 miles. About 1,300 instruments will record data on aerodynamic heating and heat transfer, structural and aerodynamic loads and the reactions of the pilot.

The article includes brief notes on the power plant and the materials of construction selected to withstand the severe conditions involved during flight. The exterior of the 'X-15' is, for example, completely sheathed in a covering of the nickel-chromium-iron alloy 'Inconel X', which will confer resistance to temperatures expected to range from −300° to 1200°F. (−185° to 650°C.). A main structure of titanium and stainless steel will reinforce the heat-resistance of the 'Inconel X', and aluminium is used for internal components which are not subjected to severe temperatures and loads. Almost 65 per cent. of the 'X-15' is of welded construction.

Nickel-plated Nickel and Copper Structures for Resistance to Thermodynamic Heating

S. ZIRINSKY and D. S. CARR: 'Heavy Electrodeposited Metallic and Flame-Sprayed Coatings in a High-Heat-Transfer Aerothermal Environment.'

45*th Ann. Tech. Proc., Amer. Electroplaters' Soc.*, 1958, pp. 97–104; disc., p. 166.

The advent of long-range ballistic missiles has confronted the design engineer with the problems posed by 'aerothermal environments' (i.e., heating induced by aerodynamic friction at high flight velocities). Re-entry into the earth's atmosphere is at a velocity which involves a temperature of thousands of degrees, high shear at the surface, and the risk of severely accelerated chemical erosion. The vehicle is exposed to such conditions for times of the order of one minute, and means must therefore be found to prevent excessive temperatures in the interior of the vehicles, for example, by the use of an external 'chemically-inert, highly-conductive heat-sink thermal shield' (a thick metallic skin, insulated from the body of the vehicle, which would absorb the heat and prevent further penetration). The thickness of material permissible in such a shield is restricted by weight considerations, and maximum increase in the *transient* thermal capacity of the heat-sink material is best achieved by allowing it partially to melt within the confines of a surface skin comprising a refractory coating made from a material selected on the basis of ease of fabrication and chemical compatibility (particularly in relation to non-solubility) with the molten heat sink.

This paper records the results of an investigation carried out to determine the suitability of various coatings for use with two typical heat-sink metals, nickel and copper.

With the copper heat-sink the following coatings were tested: nickel; nickel/platinum; nickel/palladium; nickel/rhodium; nickel/chromium; nickel/aluminium oxide; chromium; aluminium oxide. On the nickel heat-sink chromium and aluminium-oxide coatings were used. The test specimens, which were cylindrical, with a hemi-spherical leading surface, were coated with deposits of the following thicknesses:

nickel 0·005–0·04 in. (0·125–1 mm.);
rhodium 0·0001 and 0·00025 in. (0·0025 and 0·00625 mm.);
platinum 0·0001 in. (0·0025 mm.);
palladium 0·0001 in. (0·0025 mm.) and
chromium 0·005–0·035 (0·125–0·875 mm.).

The aluminium oxide was applied by flame spraying, in thicknesses in the range 0·005–0·02 in. (0·125–0·5 mm.). Full particulars are given of the plating solutions and procedures used.

The rods were tested by exposure to the plasma exhaust of a water-stabilized arc-plasma-generating unit, under conditions involving temperatures in the range 5000°–12000°F. (2760°–6650°C.) and sub-sonic velocities of between 500 and 1000 ft./sec. (150 and 300 m./sec.). The relative thermal endurance of the coatings was determined by measurement of time to melting, or to failure by other means. Heat-transfer rates were also determined. The results are presented in tabular form.

In some cases coating improved the effective heat capacity of the heat-sink metals by 30 per cent. or more. The aluminium-oxide coatings successfully survived single cycles, but cracked as a result of repeated cycling. Chromium coatings of more than 0·005 in. (0·125 mm.) thickness invariably cracked during exposure, causing early leakage of the molten material. The nickel coatings, however, appeared unchanged during the test and ultimately failed catastrophically, in a manner similar to that of an overfilled balloon. The superimposed coatings of noble metals had no effect on surface-brightness characteristics during the test.

The paucity and scatter of the data did not permit quantitative evaluation of the influence of the thickness of the coatings, though there was some evidence that increasing the thicknesses above a limit of 0·020 in. (0·5 mm.) resulted in no corresponding increase in heat capacity. (Failure in the

coatings was apparently due to the rapid volumetric expansion of the molten heat-sink metal rather than to incipient melting: ductile coatings are therefore considered more suitable for this application.) It is concluded that noble-metal films are neither deleterious nor beneficial. Insulating coatings of Al_2O_3, above a certain thickness, effectively delayed, by their own melting, penetration of heat into the heat-sink metal.

U.S. Government Specifications for Stainless Steels

'Government Specifications for Stainless Steel.'
Materials in Design Engineering, 1958, vol. 48, Oct., pp. 133, 135, 137.

A tabulated summary of information on the compositions of stainless steels covered by specifications in the U.S. MIL, Air Force/Navy Aeronautical, and Federal categories. Cognate specifications issued by the Society of Automotive Engineers (in their general and A.M.S. series) and by the American Iron and Steel Institute are correlated with the U.S. Government schedules.

High-Alloy Castings: Compositions and U.S. Standard Classifications

E. A. SCHOEFER: 'High-Alloy Castings: Specifications and Properties.'
Metal Progress, 1958, vol. 74, Dec., pp. 95–7; 96B, C, D.

This summary review of the composition and mechanical and physical properties of high-alloy casting materials is written round a table giving details of composition, properties and typical uses of materials classified under the designations standardized by the Alloy Casting Institute. Where other designations exist, the corresponding A.I.S.I. Type numbers are added. An additional table lists, with similar particulars, cast alloys for which no A.C.I. designation exists, viz., 'Hastelloy B' and 'Chlorimet 2'; 'Hastelloy C' and 'Chlorimet 3'; 'Monel'; nickel; 'Inconel'.

See also

W. T. BRYAN: 'Selection of (Cast) Alloys for Corrosion-Resistant Service', *ibid.*, pp. 102–5.

This closely related article is concerned with the factors which require consideration in selecting alloy castings for use in corrosive conditions, the influence of composition on chemical stability, the effect of heat-treatment in relation to corrosion-resisting properties, and the characteristics of typical alloys, considered in relation to their suitability for particular types of service. Throughout these notes emphasis is laid on the fact that the first consideration in selecting an alloy for such applications is a knowledge of the characteristics of the corrosive environment. 'The user of corrosion-resistant castings must realize that there is no well-defined short cut to the selection of the exact alloy for given conditions. After all the relevant variables have been studied, e.g., concentration, temperature, etc., it is often wise to make in-plant tests of a 'short-listed' number of alloys.'

'D-319': New Chromium-Nickel-Molybdenum Austenitic Steel

'New Cr-Ni-Mo Stainless 'D-319' is Approved.'
American Metal Market, Oct. 21, 1958.

In recent years the U.S. steel industry, in conjunction with the Chemical Industry Advisory Board of the American Standards Association, has agitated increasingly for production, on a commercial scale, of a modified grade of A.I.S.I. Type 316 chromium-nickel-molybdenum stainless steel, to secure increased general corrosion-resistance. The demand was usually for a higher chromium content, but stipulations as to the content of other elements were also made. Requirements varied greatly and modified steels were not, therefore, available directly from stocks. Following a survey by the Chemical Industry Advisory Board, it was evident that sufficient demand existed for a modified chromium-nickel-molybdenum steel. A special sub-committee of the C.I.A.B., working jointly with Stainless Steel Technical Committees of the American Iron and Steel Institute, finally arrived at a standard composition which incorporated the general requirements brought to light by the survey. The background to the development of the steel, to which the designation 'D-319' has been assigned by the A.I.S.I., is described in this short note. The composition range is given below, together, for purposes of comparison, with that of Type 316.

Pending the result of a tonnage survey scheduled for 1959 the steel is known as a 'development' material. When sufficient demand develops the steel will be designated 'A.I.S.I. Type 319'.

Formal approval for the use of 'D-319' in Code construction has been obtained through Special Ruling Case No. 1254 issued by the Boiler and Pressure Vessel Committee of the American Society of Mechanical Engineers. Enquiry has determined that substantially all the principal stainless-steel

Steel	C %	Si %	Mn %	S %	P %	Cr %	Ni %	Mo %
'D-319'	0·07 max.	1·0 max.	2·0 max.	0·03 max.	0·045 max.	17·5–19·5	11·0–15·0	2·25–3·0
A.I.S.I. Type 316	0·08 max.	1·0 max.	2·0 max.	0·03 max.	0·045 max.	16·0–18·0	10·0–14·0	2·0–3·0

producers in America have stocks of 'D-319' steel immediately available, some in ingot, others in finished, form.

Specifications for Nickel-containing Materials

See abstract on p. 43.

Forms of Nickel available as Alloying Additions for Steels

See abstract on p. 48.

Influence of Lithium Additions on the Properties of Austenitic Steel

R. P. ZALETAEVA: 'Effect of Lithium on Certain Properties of an Austenitic Steel.'

Metallovedenie i Obrabotka Metallov, 1957, No. 9, pp. 25-27.

The influence of lithium on the gas content, inclusion content, density, hot-workability, creep behaviour and room- and elevated-temperature mechanical properties was determined for an austenitic steel of the following composition: carbon 0·15 max., silicon 0·8, manganese 0·4-0·8, sulphur 0·03 max., phosphorus 0·035 max., nickel 13-15, chromium 13-15, molybdenum 0·45-0·6, tungsten 2-2·75, per cent.

The lithium was added in the form of silicon-lithium, iron-silicon-lithium and lithium-aluminium alloys, a procedure which, compared with the effects of introducing the metal in elemental form, minimized oxidation and spattering, without lessening the effectiveness of the lithium. Of the alloys investigated, a low-lithium iron-silicon-lithium alloy proved the most suitable addition agent. The composition of the alloys was designed to produce a lithium content of 0·01-0·02, 0·03-0·05 or 0·1 per cent. (0·2-0·4, 0·6-1, and 2·0 lbs. per ton) in the steel.

Treatment with lithium refined the ingot structure and narrowed its zone of columnar crystals: optimum results were obtained with additions of 0·03 per cent. and above. Austenite in untreated steel was coarser than that in treated specimens.

Data illustrating the effect of lithium on the oxygen, nitrogen and hydrogen contents of the steel show that all were lowered by the addition of lithium: the most potent effect resulted from treatment with 0·03 per cent. lithium: larger amounts were of no further advantage in this respect. The quantity of non-metallic inclusions was reduced by an addition of 0·03 per cent. lithium, even when the amounts initially present were relatively low. The density of the treated steel increased, probably as a result of de-gassing.

Tests to determine the comparative hot-workability of treated and untreated specimens showed the treated steel to be more ductile and, in upsetting, cracks occurred at a somewhat greater reduction in height than in specimens of untreated steel. Similarly, the tensile strength and ductility of lithium-treated steel at 20°C. was better than that of untreated steel, and rupture strength was also improved: data on these properties are tabulated.

Lithium had no effect on the oxidation-resistance of steel exposed for 24 hours at temperatures of 900° and 1100°C.

Stress-Corrosion Cracking in Austenitic Steels

L. R. SCHARFSTEIN and W. F. BRINDLEY: 'Chloride Stress-Corrosion Cracking of Austenitic Stainless Steel: Effect of Temperature and pH.'

Corrosion, 1958, vol. 14, Dec., pp. 588t-92t.

Until some 15 years ago reports of the incidence of stress-corrosion cracking in austenitic stainless steels were rare, but the recent increased use of these materials has brought evidence of their susceptibility to this type of failure, particularly in aqueous chloride solutions. (Reference is made to typical reports of failures in chloride-contaminated water at elevated temperatures.) The fact that relatively few cases of such failure have been reported as occurring in dilute chloride solutions at temperatures below the boiling point of water indicates, however, that lowering of the temperature of corroding media of that type must lessen susceptibility to attack. The aim of the research described in this paper was, therefore, to determine susceptibility in dilute sodium-chloride solutions between the temperatures of 165° and 200°F. (75° and 95°C.). Stressed U-bend specimens (illustrated) were used, as having proved suitable in other studies of stress-corrosion cracking and therefore being capable of providing data directly comparable with results already obtained with such specimens at 500°F. (260°C.).

The materials subjected to test comprised chromium-nickel steels of the 18-8 and 18-8-Nb grades (A.I.S.I. Types 304 and 347), both in the annealed condition, and the unstabilized Type 304 steel in the sensitized condition.

Experimental procedure, inhibitory addition media added to the solutions, methods of inspection, and results are described in detail.

The behaviour of the specimens in the range of conditions studied indicates that cold-worked and stressed austenitic stainless steels are susceptible to chloride stress-corrosion cracking at temperatures as low as 165° to 200°F. (75° to 95°C.), when as little as 5 p.p.m. of chloride is present in the water. Little difference was found between the resistance of Types 304 and 347 steels, except with the sensitized Type 304. Sensitized steel cracks predominantly *inter*granularly and exhibits longer and deeper cracks than the unsensitized material. The latter cracks *trans*-granularly. (Typical photomicrographs are shown.)

Increasing concentration of dissolved oxygen was found to have a slightly deleterious influence.

Increase in pH (controlled, in most tests, at 6-8·5 or 10·6-11·2) proved beneficial, an effect which is attributed to reduction of pitting in the aqueous chloride solution. The addition of alkaline phosphate gave results demonstrating the inhibiting property of the phosphate ion: a concentration of alkaline phosphate equimolar with the chloride-ion concentration conferred complete protection from both

cracking and pitting in all the stainless steels tested. The phosphate ion is, however, an unsafe inhibitor for the sensitized steel, because an insufficient concentration results in localized pitting attack.

The addition of sodium hydroxide or potassium chromate was also beneficial: these results are interpreted as illustrating the inhibiting influence of increased hydroxyl ion.

The authors conclude that chloride stress-corrosion cracking of austenitic stainless steel in the range 165°-200°F. (75°-95°C.) is much less severe than that experienced at 500°F. (260°C.), and that at these lower temperatures it can occur only in specific conditions.

See also

J. P. HUGO and L. G. NEL: 'Failure of Type 316 Stainless-Steel Autoclave Components.'

Corrosion, 1958, vol. 14, Dec., pp. 553t-6t.

The authors describe investigation of the cause of failure, after only short periods of service, of autoclave components in 18-8-Mo steel of A.I.S.I. Type 316. (Body and cover were of 'Monel': cooling coil, thermowell, sampling tube, valve and associated fittings were of the stainless steel.) The specification met by the material was that of suitability for service at a maximum temperature of 343°C. with maximum pressure of 2500 p.s.i. (1·1 t.s.i.; 1·75 kg./mm.^{2}) and the manufacturers considered the autoclave to be suitable, in such conditions, for handling sodium hydroxide up to 50 per cent. concentration. In order to provide an additional margin of safety, however, operating conditions for the autoclaves were fixed by the users at 300°C. and at pressures ranging, at maximum temperature, from 1200 to 1500 p.s.i. (0·5-0·7 t.s.i.; 0·8-1 kg./mm.^{2}), depending on the liquid charge. Precautions adopted to ensure consistent maintenance of such conditions are recorded.

Operational life was, however, only between 38 and 52 hours. (Failure occurred during the night and since pressure-time records were not kept, the exact instant of failure could not be determined.) On dismantling, it was found that failure had occurred in some of the nuts. Two typical examples are described, with details of the location from which the nuts were taken and of the position and nature of the cracking found in them. A small portion cut from the cooling coil also revealed cracks. (It is noted that ultrasonic dye-penetrant and radiographic testing of the 'Monel' autoclave body failed to reveal cracks or defects of any nature, and that the 'Monel' cover also proved to be free from defects.)

The cracks in all the steel specimens examined were found to be transgranular and showed a tendency to be branched and blunt: a black corrosion product was detected in the cracks. The surfaces of the components showed little, if any, evidence of pitting, and the origins of the cracks did not appear to coincide with the pits.

The authors conclude, after considering all the factors involved, that the failure of these components, which were operating in vapour-phase conditions, is attributable to stress corrosion. They believe the corroding medium to have been chloride-contaminated steam. Results of work by WILLIAMS and ECKEL (*Jnl. Amer. Soc. Naval Engineers*, 1956, vol. 68, pp. 93-104: *Nickel Bulletin*, 1956, vol. 29, No. 6, p. 114) and others are advanced in support of this view. It is suggested that the stresses which caused failure must have been predominantly residual in character, particularly in the case of the cooling coil. In the nuts, existing residual stresses would appear to have been intensified by externally applied stress. There is evidence that the components had undergone some cold working, e.g., in machining or fabrication, prior to installation. It is concluded that there was no indication of the use of excessive operating temperatures or pressures.

Corrosion by Hydrogen Sulphide

E. B. BACKENSTO and J. W. SJOBERG: 'New Hydrogen-Sulphide Corrosion Curves.'

Petroleum Refiner, 1958, vol. 37, Dec., pp. 119-20.

Recent laboratory investigation by the Socony Mobil Oil Company has resulted in revision and extension of published data for corrosion by hydrogen sulphide. Two new charts published in this article are of immediate interest in relation to corrosion of steels in re-formers employed in petroleum catalytic refining processes which utilize hydrogen.

The two charts show maximum rate of corrosion to be expected over a wide range of hydrogen-sulphide concentration and temperature encountered in petroleum catalytic refining processes in which hydrogen is used. Chart 1 relates to chromium steels (in most cases up to 5 per cent. chromium, but in many cases applicable also to steels containing up to 9 per cent. chromium); chart 2 relates to austenitic chromium-nickel steels. The major variables plotted are temperature and hydrogen-sulphide concentration, with curves showing constant corrosion rate. The pressure variable is confined to the range 175-500 p.s.i.g. (0·12-0·35 kg./mm.^{2} above atmospheric pressure) because data relating to 0·1 per cent. hydrogen sulphide by volume and 985°F. (530°C.) indicate that the rate of corrosion is unaffected by pressure within that range.

The iso-corrosion rate curves represent an interpretation of results obtained in laboratory tests of more than 150 hours' exposure. The values shown on the charts have been confirmed by tests in various commercial re-forming units.

Corrosion of Nickel in Sulphuric Acid

See abstract on p. 39.

Chromium-Nickel-Steel Castings for Nuclear Power Applications: Specification

G. SORKIN: 'Steel Castings for Radioactive Service.'

Foundry, 1958, vol. 86, Oct., pp. 70-3.

MIL-S-18262 (*SHIPS*), issued by the *U.S. Bureau of Ships* on Nov. 10, 1954, was the first military specification to lay down requirements for corrosion-resisting steel castings intended for use in systems

involving exposure to radioactivity. The exclusive use of wrought material in the U.S. nuclear-powered submarine 'Nautilus' reflected the initial reluctance of designers of nuclear power plants to specify castings for critical applications. Since that time much experience has been accumulated in the purchase and inspection of steel castings for such service, and the requirements of the original specification have been modified in the light of the new information. The specification in its revised form (*MIL-S-18262A (SHIPS)*, dated Feb. 7, 1958) is the subject of the present article.

The specification now relates mainly to machined castings in finished form, although it may be used also for ordering castings which are to be subsequently machined or otherwise finished. Ranges of composition and mechanical properties stipulated for the four grades of steel covered are given in Table I. The restrictions on the amount of tantalum and cobalt present are necessary due to the radiation risks involved.

The castings, which are used in an all-welded installation permitting no normal maintenance inspection, are subjected in service to conditions involving high-temperature water at a pressure of 2000 p.s.i., thermal and pressure cycling, nozzle pipe stress, mechanical shock and bending moments. Quality requirements are therefore severe.

In addition to laying down cleaning procedure and heat-treatment and stipulating resistance to intergranular corrosion, the specification embodies standards for radiographic and hydrostatic testing and for liquid-penetrant examination. To reduce the cost of radiographic examination, the concept of 'identified casting series' is introduced; i.e., a grade of castings qualifying for modified radiographic inspection by virtue of the fact that the purchaser and foundry designate the 'critical areas' where defects in excess of the radiographic standards can be anticipated. The provisions of the specification relevant to this aspect and those relating to penetrant examination are quoted by the author. Reference is also made to permissible methods of repairing defects.

Nickel-Alloy Steels for use in the Petroleum Industry

S. BARRACLOUGH: 'Wrought Steels for the Petroleum Industry.'

Inst. Petroleum Rev., 1958, vol. 12, Nov., pp. 370-4.

The increasingly international character of the oil industry and the predominance, until recently, of plant of American origin, have made it imperative that equipment emanating from different sources should be freely interchangeable in all parts of the world. Confronted with this problem, the steel and petroleum industries and allied equipment manufacturers have, under the aegis of the British Standards Institution, combined forces to provide specifications standardizing requirements for steels used in petroleum engineering. (In this connexion, reference may be made to *B.S.* 1501-1506 : 1958, which lays down requirements for carbon, low-alloy and stainless steels intended for use in the petroleum industry: see abstract in *Nickel Bulletin* 1958, vol. 31, No. 12, p. 348.) Applications and service conditions in the petroleum industry vary greatly, necessitating use of an extremely wide range of steels to ensure selection of the most suitable material for a specific application. The author has therefore made no attempt to cover the entire field, but has confined his review to discussion of the salient characteristics of steels which have proved their worth sufficiently to justify recognition as standard types.

The steels are discussed in three sections, relating

Table I

Composition and Properties of Stainless-Steel Castings for Nuclear Power Applications

Class	Composition											Mechanical Properties		
	C max. %	Si max. %	Mn max. %	S max. %	P max. %	Ni %	Cr %	Mo %	Nb %	Ta max. %	Co* max. %	Tensile Strength (min.)	†Yield Strength (min.)	Elongation in 2 in. (min.)
I	0·08	2·0	1·5	0·045	0·045	8-11	18-21	—	—	—	0·2	70,000 p.s.i. 31 t.s.i. 49 kg./mm.²	28,000 p.s.i. 12·5 t.s.i. 19·5kg./mm.²	35%
II	0·08	2·0	1·5	0·045	0·045	9-12	18-21	—	8xC-0·85 max.	0·1	0·2	70,000 p.s.i.	30,000 p.s.i. 13·5 t.s.i. 21kg./mm.²	30%
III	0·08	2·0	1·5	0·045	0·045	9-12	18-21	2-3	—	—	0·2	70,000 p.s.i.	30,000 p.s.i.	30%
IV	0·08	2·0	1·5	0·035	0·045	12-15	22-26	—	10xC-1·0 max.	0·1	0·2	70,000 p.s.i.	28,000 p.s.i.	30%

* Unless otherwise specified.

† Determined by use of the 0·2% offset method or by a total extension under load of 0·3%, using an extensometer reading to 0·0001 in. per in.

to (1) low- and medium-carbon steels; (2) low-alloy steels; (3) high-alloy steels.

By far the greatest tonnage of steel used in the oil industry is represented by carbon steel. This fact is emphasized by reference to typical high-tensile steels, to those developed to provide a specific impact-resistance at low temperature, and to the relative creep-resistance of steels deoxidized by addition of silicon or killed with aluminium. The section relating to low-alloy steels is introduced by mention of the influence of alloying elements on the properties obtained and by a table, covering various nickel, nickel-chromium and nickel-chromium-molybdenum steels, which records the limits of section-size within which specific tensile strengths are obtainable. Steels suitable for case-hardening are discussed, but the greater part of the section is devoted to consideration of steels which have proved suitable for low- or elevated-temperature applications. Some consideration is given to the rôle of alloy content in connexion with the suitability of such steels for use in specific applications.

The term 'high-alloy steels' is regarded in this article as embracing those of 18-8 chromium-nickel austenitic type. Mention is made of failure by weld decay and its prevention by stabilization with titanium or niobium. Reference is also made to the increased resistance to attack by certain types of corrosive media which is conferred by addition of molybdenum. Control of molybdenum content, necessary to avoid risk of embrittlement by sigma phase at high temperatures, is briefly considered.

Applications of High-Nickel Alloys in the Petroleum Industry

G. L. SWALES: 'Some Applications of High-Nickel Alloys in the Petroleum Industry.'
Petroleum, 1958, vol. 21, Nov., pp. 391–4; Dec., pp. 439–43.

The wide range of applications found by nickel-containing materials in the petroleum industry is well illustrated by the use, in a recently constructed American refinery, of more than one million pounds of nickel. Whilst a large proportion of this total is represented by the nickel content of austenitic stainless steels and copper-base alloys, a significant percentage is accounted for by the nickel-base alloys used to combat the severely corrosive conditions which obtain in practically every phase of operations, from oil production to refining processes. In this context, the present paper reviews the problems which confront the plant designer, and gives examples of applications for which various nickel-base alloys have proved their worth.

The survey may be conveniently divided into four parts: (1) applications in oil-well equipment; (2) applications in the fundamental distillation processes; (3) corrosion problems involved in specific refinery processes and materials employed to overcome them; and (4) high-temperature applications of high-nickel alloys in the petrochemical industry.

In petroleum engineering the nickel-copper alloy 'Monel' is the most widely used of all the high-nickel alloys: oil production is discussed by the author solely in relation to the use of the various grades of this alloy as a material of construction. Reference is first made to the results of a field study by The International Nickel Company, Inc., carried out to find a means of conferring corrosion-resistance on the steel piling of off-shore drilling platforms, particularly in the tidal and splash zones. Sheathing the steel columns with 'Monel' was found to ensure excellent resistance to such conditions: alternatively, the employment of clad steels has been studied. The use of the precipitation-hardenable alloy 'K Monel' for non-magnetic drill collars also marks an important advance in oil-well surveying: the relevant properties of the alloy and the advantages which have accrued from its use are outlined. The first section of the survey ends with a short review of the applications of 'Monel', the cast grade 'S Monel', and the heat-treatable 'K Monel' (e.g., for valves, pumps, gas-lift equipment, sucker rods, polished rods, etc.) in oil wells operating under extremely corrosive conditions.

Corrosion encountered in crude-oil distillation is due mainly to the presence of (1) hydrochloric acid formed by the hydrolysis of chlorides contained in the crude; (2) sulphur compounds such as mercaptans and hydrogen sulphide; (3) naphthenic acid in some crudes, particularly in Venezuelan and Californian types. The problems involved in such conditions, and the plant in which they occur, are discussed. 'Monel' exhibits outstanding resistance to environments involving exposure to mineral acids formed by hydrolysis of chlorides, but at temperatures in excess of 500°F. (260°C.) this alloy suffers severe intergranular attack in the presence of organic sulphur compounds. To combat corrosion in crude columns processing corrosive feed stock it is common practice to employ steel clad with 'Monel', or to use the alloy as an applied lining for the upper portions of the shell and also for the upper trays and bubble caps, whilst 13 per cent. chromium steel or 18-8 chromium-nickel steel is chosen for the lower-high-temperature zones. Corrosion by naphthenic acids occurs at temperatures above 500°F. (260°C.), chiefly in atmospheric and vacuum distillation units: in such conditions the molybdenum-containing stainless steels and 'Inconel' have proved their value as materials of construction. In catalytic cracking, re-forming and hydro-desulphurization processes 'Monel' and the nickel-base hard-surfacing alloy 'Colmonoy' find useful application in handling corrosive condensates and wash waters.

The third section of the review is devoted to discussion of refinery processes, describing some corrosion problems which can be countered only by careful selection of materials of construction in the light of their behaviour under specific service conditions. In each case the author gives a brief summary of the operational conditions, mentions the plant where risk of corrosion is particularly likely, and reviews the nickel-containing materials

which experience has shown to give optimum corrosion-resistance. Reference is made below to the processes surveyed and the alloys considered.

Hydrofluoric-Acid Alkylation, in which olefines are united with *iso*butane, to form high-octane aviation-blend stock. Concentrated hydrofluoric acid is used as a catalyst. 'Monel' or 70-30 cupro-nickel is used to resist the most severe conditions.

Sulphuric-Acid Alkylation and Acid Treatment of Distillates, involving handling of sulphuric-acid liquors and sludges, over a wide range of temperatures and concentrations. The corrosion-resistance and resultant applications of the following materials are briefly discussed: complex nickel-chromium-molybdenum steels, such as 'Carpenter 20', 'Durimet 20', 'Alloyco 20', 'Paralloy 20', 'Langalloy 20V', 'Worthite'; the nickel-molybdenum-base alloys 'Hastelloy B' and 'C' and 'Corronel B'; the nickel-base alloys 'Monel', 'K Monel', 'S Monel' and 'Hastelloy D'; and the nickel-chromium-molybdenum alloy 'Ni-O-Nel'.

Isomerization, using a catalyst based on anhydrous aluminium chloride, with hydrochloric acid as a catalyst promoter. 'Hastelloy B', 'Corronel B' and 'Monel' are considered in this connexion.

Amine Gas Treatment, for removal of H_2S and CO_2 from refinery hydrocarbon gas streams. The 'Girbotol' process and glycol amine gas treatment are discussed. Depending on the specific nature and severity of the conditions, 'Monel', 'Inconel', 18-8-type stainless steels and carbon steels are used for the plant.

Caustic Treatment with caustic-soda or -potash solutions containing solubility promoters, forms the basis of a number of commercial processes (e.g., removal of mercaptan sulphur from petroleum products). 'Monel' and, at high temperatures, 'Inconel', find application in the plant concerned.

Furfural Treatment of Lube Oils. 'Monel' has found application as linings in the furfural-recovery section.

It is anticipated that increasing demands for hydrogen in refining and associated petrochemical operations will result in more extensive production of the gas by steam-reforming refinery gases such as propane. The paper therefore includes also a review of the conditions to which furnace tubes, ancillary piping, and tube supports are exposed at the temperatures (1900°F.; 1040°C.) used in the process. The suitability of the nickel-chromium-base alloy 'Inconel', the nickel-chromium-iron alloy 'Incoloy', and 25-20 chromium-nickel steel for such applications is assessed on the basis of their high-temperature properties, metallurgical stability, and resistance to the various environments encountered on the combustion and product sides of the furnace (i.e., resistance to attack by sulphur, resistance to carburization and to oxidation).

Finally, reference is made to the use of 'Inconel' or the nickel-chromium-iron-silicon alloy 'Nimonic DS' for flare-stack tips, and to the excellent resistance of these materials to the severe conditions of temperature and stress induced by exposure to winds of high velocity.

Machining and Assembly of the 'Rotodyne' Rotor Blade

See abstract on p. 48.

Nitrogen as a Weld-Backing Gas

R. V. ANDERSON: 'The Value of Nitrogen as a Weld-Backing Gas.'

Welding Jnl., 1958, vol. 37, Dec., pp. 1193-5.

The article is introduced by a concise summary of the effects of oxygen, hydrogen and nitrogen in weld metals, and of the reasons for the use of inert gases as shielding media. This is followed by a report of experiments in the welding of 'Nimonic 75', a high-chromium stainless steel, and an austenitic chromium-nickel-molybdenum steel, using (1) argon gas for the torch and back-up, and (2) argon gas for the torch and nitrogen for back-up. All three materials were welded in various thicknesses. The immediate interest of the experiments was development of a back-up method alternative to the use of argon, in fabrication of components of missile assemblies. The change from argon to nitrogen would have considerable economic advantage.

Results of the tests are detailed in terms of the nitrogen content of the base metal and of the filler wire, and of the nitrogen content of the welds (1) argon-backed, and (2) nitrogen-backed. The behaviour of the welds under tensile test, X-ray examination, bend tests and photomicrographic examination is also reported.

The tests show that the high-nickel alloy and the martensitic stainless steel are relatively insensitive to pick-up of nitrogen backing gas. The austenitic stainless steel is slightly more sensitive, but not to an extent forbidding use of this method for such material. The work indicates that nitrogen may safely be introduced into the weld metal at a maximum level of 1/100th per cent. for each 1 per cent. of chromium present in the parent metal.

Resistance-Welded Nickel-Alloy Tube Joints in Heat Exchangers

J. J. MUELLER: 'Resistance-Welded Tube Joints in Heat Exchangers.'

Metal Progress, 1958, vol. 74, Dec., pp. 106-11.

Heat exchangers working under modern conditions are called upon to operate at a much higher level of efficiency than in the past. In addition, they must provide trouble-free service for much longer periods, at higher temperatures and in more corrosive environments. Such changes have made more stringent the requirements for tube-to-tube sheet joints, and considerable trouble has been encountered in this connexion.

This article records experimental development, by the Martin Company, Baltimore (in conjunction with Oak Ridge National Laboratory and the U.S. Navy Bureau of Ships) of procedure for resistance-welding of tube joints in heat exchangers. The

design and methods initially tried are described and illustrated, and details are given of the design of joint and the resistance-welding procedure finally adopted as consistently providing sound uniform joints, capable of resisting severe thermal-shock cycling. Inspection tests used for evaluation of quality of joint are also reviewed: these comprise metallographic examination, hydrostatic pressure test and thermal-shock tests.

The procedure eventually standardized involves use of a drilled and reamed tube sheet and a flared tube. The edge of the tube sheet hole is chamfered, to eliminate sharp corner contact between the components. Welding is performed with a conical electrode of particular design, which is illustrated in the article. Methods for welding tube clusters are also described.

Joining of 'Zircaloy' to Austenitic Stainless Steel

J. B. McANDREW, R. NECHELES and H. SCHWARTZBART: 'Joining of 'Zircaloy' to Stainless Steel.'

Welding Jnl., 1958, vol. 37, Dec., pp. 529s-34s.

Niobium-stabilized stainless steel (A.I.S.I. Type 347) and the zirconium-base alloy 'Zircaloy' both have properties which make them suitable for use in construction of pressurized-water nuclear-power reactors. In certain applications involving the use of heavy-walled tubing it is necessary to make a joint between the two alloys, and service conditions make it essential that the joint should function with a high degree of reliability over a long period, provide a high-pressure seal, and be resistant to corrosion in an environment of high-temperature water. This paper reports a study of brazing and welding techniques investigated as possible methods of producing joints satisfying such a specification.

Difficulties were anticipated due to: (1) the poor mechanical and corrosion-resisting properties of high-Zr zirconium - iron alloy, which completely excluded the possibility of ordinary fusion welding and added to the difficulties involved in other welding methods; (2) the large difference between the thermal-expansion coefficients of the two materials; and (3) lack of filler materials which would be capable of producing brazed joints having adequate corrosion-resistance.

Attention was directed first to the achievement of corrosion-resistant metallurgical seals, as being a primary essential. Critical consideration led to the study of (*a*) flash-butt welding, and (*b*) brazing, as possible methods. The test criterion adopted was exposure to static distilled water at 680°F. (360°C.) for 1200 hours. The tests were intermittent, in that specimens were removed from the autoclave for examination at intervals varying from a few hours to several hundred hours. Specimens were removed from test only after cumulative exposure of at least 1200 hours or if complete separation of the joint members had occurred. The condition of specimens which survived the test without total failure was evaluated by breaking the joint apart and observing the fracture surfaces.

Three types of welded joint and four varieties of brazed joint were made: details are given of design and of methods of preparation and joining.

Brazing

The experimental brazing alloys were of the silver-, aluminium-, gold-, copper-, niobium-, nickel-, palladium-, tin- and zirconium-base types: nominal compositions of 55 filler materials are given. Many of these were eliminated from further consideration as a result of poor performance in an initial corrosion test, but encouraging results were obtained with alloys of the following types:

Cu + 20% Pd + 3% In
Ni + 20% Pd + 10% Si
Ni + 30% Ge + 13% Cr
Ni + 40% In
Ni + 7% P
Zr + 5% Be
Zr + 15% Pd + 5% Nb
Zr + 30% Fe + 10% Mn
Zr + 10% Fe + 10% Cr

Tests with these alloys and with various types of joint showed that the incidence of thermal stress superimposed on the effects of the corrosive environment is a factor of considerable significance. The Zr + 5% Be alloy emerged as the only brazing medium which gave acceptable results.

The characteristics and behaviour of the respective joints and their corrosion-resistant qualities are reported by means of tabulated results and photographs of typical joints broken after exposure to the corrosion test.

Welding

Flash-butt welding experiments were made with joints of varying geometry, in thick-walled tubing. Although some of the results were promising, the condition of these joints after corrosion test (illustrated) confirms the significance of thermal stress (particularly in flanged joints) as a problem which will require further study.

The general conclusion drawn is that 'the occasionally good results obtained with both flash-butt welding and brazing, even with unfavourable joint geometry, indicate that either method might be useful for some applications, but that the quality of the joint obtained will depend on the thermal stresses involved in the individual joint designs. Brazing is to be preferred, since this method permits greater flexibility of design. The zirconium-beryllium alloy is judged to be the best of those investigated. Whether there is a toxicity hazard in the use of this alloy has not been determined, but suitable precautions should be taken on the assumption that there is such danger until the contrary has been proved.'

Nickel-Chromium-Boron and Gold-Nickel Brazing Alloys

W. H. CHANG: 'Further Evaluation of Ni-Cr-B and Au-18Ni Brazing Alloys.'

Welding Journal, 1958, vol. 37, Dec., pp. 535S-42S.

In an earlier paper the author reported investigation of the basic characteristics of five heat-resisting brazing alloys, evaluated on the basis of room-temperature tensile tests and microscopical examination (*ibid.*,

1956, vol. 35, pp. 431S-3S; *Nickel Bulletin*, 1957, vol. 30, No. 1-2, p. 25).

In order to assess the usefulness of these alloys for elevated-temperature applications, further tests were made on two alloys of the following composition:

(1) Cr 15, Si 4, B 3·5, C 1, Fe 4, per cent., bal. Ni. (approx. liquidus 1925°F.; 1050°C.)
(2) Au 82, Ni 18, per cent. (approx. liquidus 1742°F.; 950°C.)

The nickel-chromium-boron alloy typifies multi-phase nickel-chromium-base alloys, in that the mechanical properties of the brazed joints deteriorate with increasing joint clearance, due to the appearance of brittle phases. The gold-nickel alloy is a solid-solution type, of which the basic structure, and hence the mechanical properties, do not vary appreciably with joint clearance. The basis metals used were stainless steels of the high-chromium (A.I.S.I. Type 410) and chromium-nickel-niobium (A.I.S.I. Type 347) grades. Joints made in these materials were subjected to tensile, impact and oxidation tests, over a range of temperature.

All the specimens were of the butt-joint design; for details of design and brazing procedure, reference is directed to the earlier paper. In the experiments now described, the joints brazed with the gold-nickel alloy had a clearance of either 'zero' or 0·004 in. (0·1 mm.), whereas those brazed with the nickel-base alloy all had a clearance of 'zero'. The brazing cycles were 2200°F. (1204°C.)/10 min. for the nickel-chromium-boron alloy and 2000°F. (1093°C.)/10 min. for the gold-nickel alloy.

Since the brazing temperatures were above the A_1 temperature of the chromium steel, specimens prepared from this material were hardened, as a result of brazing, from the as-received Rockwell A_{60} to Rockwell C_{40}. Some of the tensile specimens of this steel were tested in the as-brazed condition, but the majority were tempered at 1200°F. (650°C.) for 3 hours after brazing, a treatment which restored the hardness and tensile properties of the brazed specimens to a level approximately equal to those of the as-received base metal.

Testing

Standard 0·250 in. (0·625 mm.) tensile bars machined from the brazed specimens were tested at room temperature and at 500°, 800°, 1000° and 1200°F. (260°, 425°, 540° and 650°C.). For the oxidation tests, as-brazed tensile-bar specimens of the chromium-nickel steel were exposed in a muffle at 1500°F. (815°C.) for 1, 2, 4 or 8 weeks, with air circulating at a rate of 10 cu. ft. (0·28 cu. m.) per hour. Oxidation was assessed by microscopic examination, for determination of depth of oxidation, and by tensile test, to estimate the loss of strength at room temperature. Impact tests, at room temperature and at 1200°F. (650°C.), were made on standard Charpy V-notch specimens with the root of the notch contained in the brazed joint.

A comprehensive report of the results of the respective tests is presented, in tabular and graphical form, supplemented by representative photomicrographs. Each section of the report is the subject of detailed discussion.

An all-over consideration of the behaviour of brazed joints leads to the following conclusions:

The tensile strength of butt joints of the austenitic steel brazed with the nickel-chromium-boron alloy at 'zero' clearance is practically equal to that of the base metal over the temperature range 75°-1200°F. (24°-650°C.).

The strength of joints in the austenitic steel brazed with the gold-nickel alloy is equal to that of the base metal up to 600°F. (315°C.) for the 'zero' joints, and up to 800°F. (425°C.) for the joints made with 0·004-in. (0·1 mm.) clearance. Above these temperatures the joints become weaker than the base metal, the effect being more pronounced at 'zero' than at 0·004-in. (0·1 mm.) clearance. The better hot strength at the larger clearance is attributed to the strengthening of the gold-nickel alloy by dissolution of the base metal.

Rate of dissolution of the high-chromium steel by the nickel-chromium-boron alloy appears to be more rapid than the rate of diffusion of the brazing alloy into the base metal: this results in the retention of brittle phases in the 'zero' joints.

Untempered chromium-steel joints brazed with gold-nickel alloy at 0·004-in. (0·1 mm.) clearance show strength of more than 190,000 p.s.i. (84·8 t.s.i.: 133·5 kg./mm.^{2}) at temperatures below 500°F. (260°C.). After tempering, these joints, regardless of clearance, have tensile properties approximately equal to those of the base metal, over the entire range of temperatures used in these tests.

The austenitic-steel butt joints brazed with either of the brazing alloys used showed excellent impact-resistance: the values were higher at 1200°F. (650°C.) than at room temperature. The joints brazed with the gold-nickel alloy exhibited higher impact values at 'zero' than at 0·004-in. (0·1 mm.) clearance.

The chromium-steel joints brazed with nickel-chromium-boron alloy at 'zero' clearance showed poor impact values, due to the presence of brittle phases, but the values for the joints brazed with gold-nickel alloy proved satisfactory at 'zero' clearance and excellent at 0·004-in. (0·1 mm.) clearance. The effects of clearance on impact properties are explained in terms of the toughness of the base metal relative to that of the brazed joint.

ANALYSIS

Determination of Nickel in Sodium

See abstract on p. 39.

Attention is drawn to the fact that many of the names of materials mentioned in The Nickel Bulletin are *Registered Trade Marks*.

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